Organic & Biomolecular Chemistry

Supporting Information

Assembly of isoxazol-5-one with 2-unsustituted imidazole *N*-oxides and aldehydes

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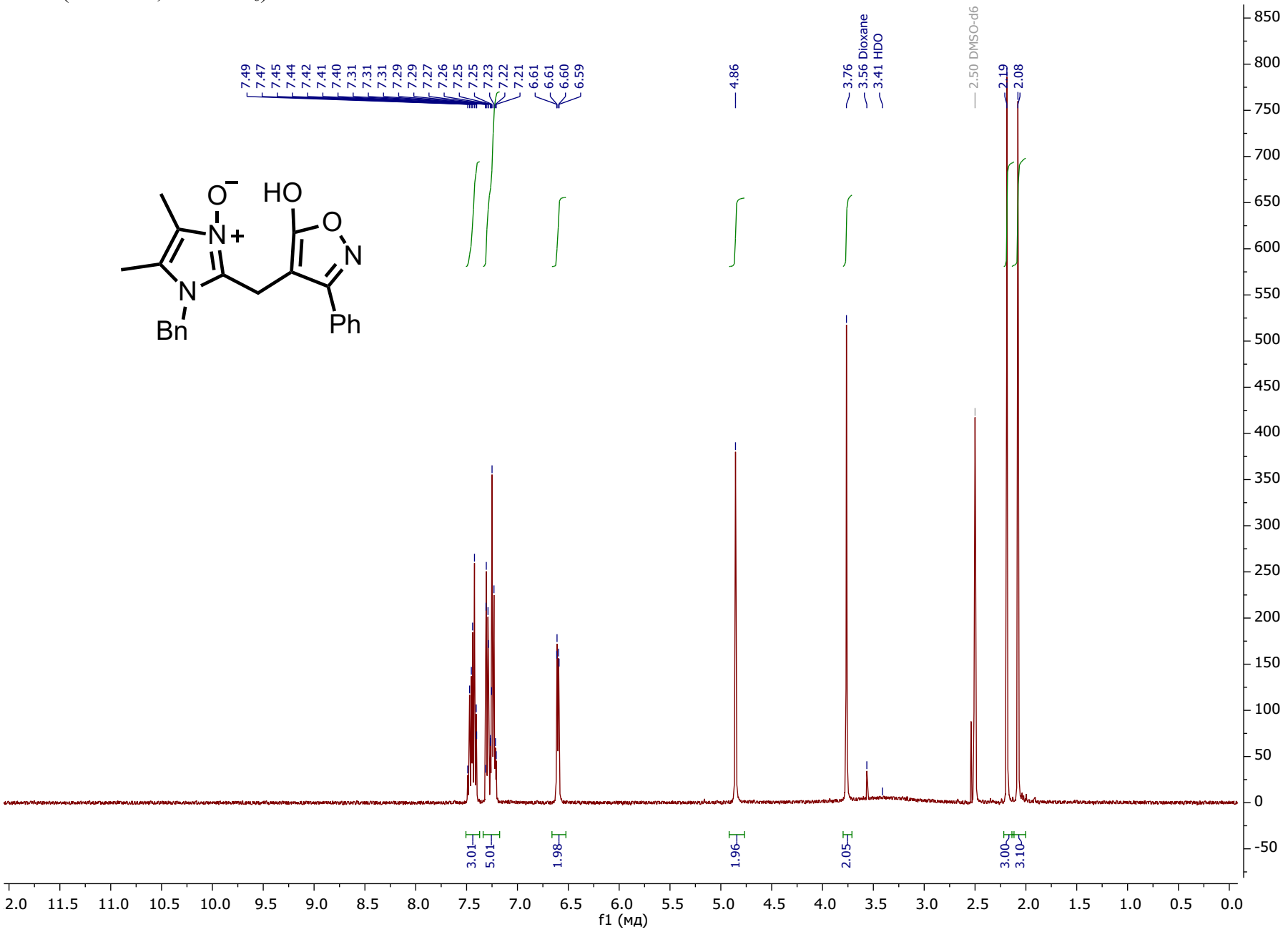
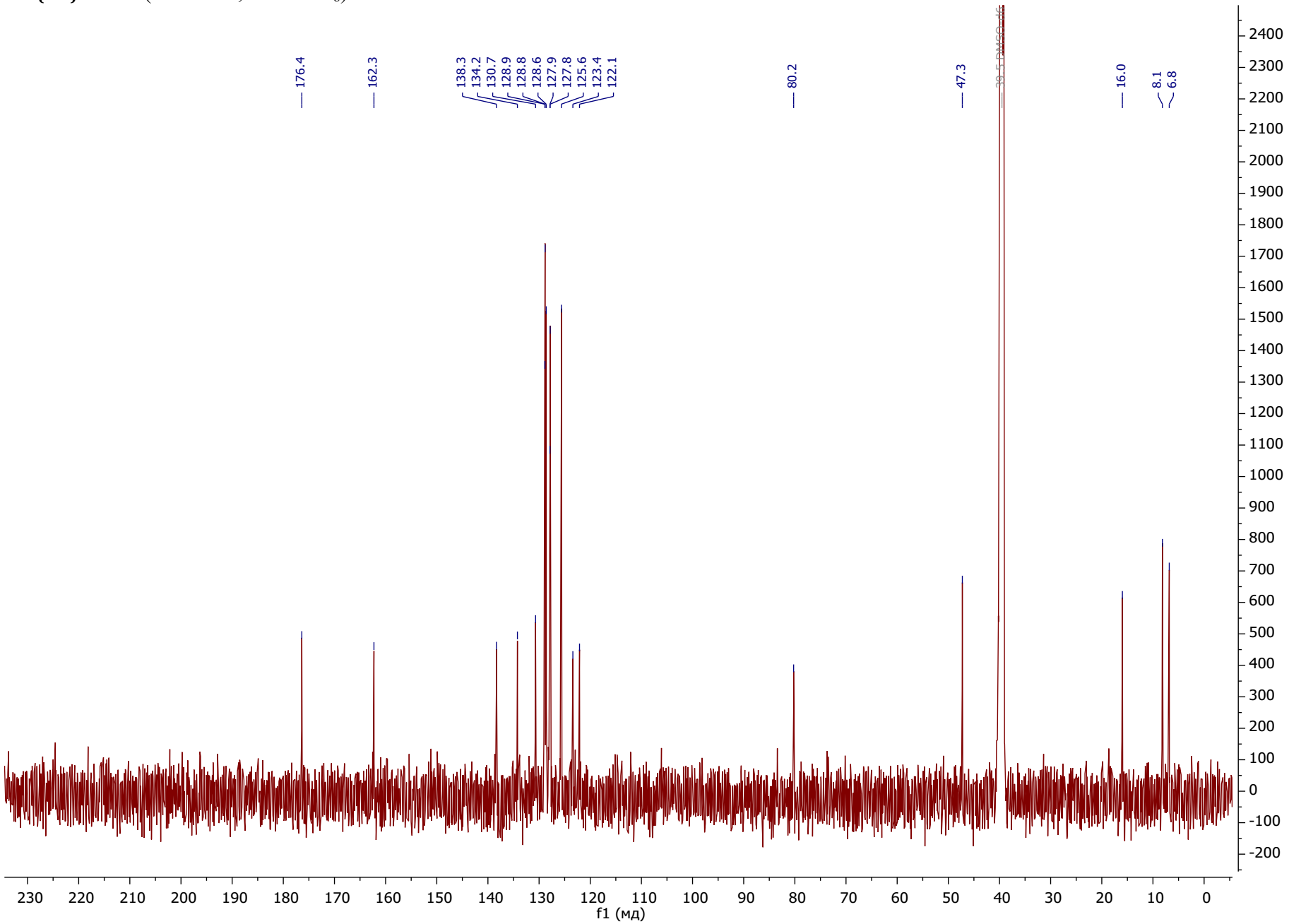
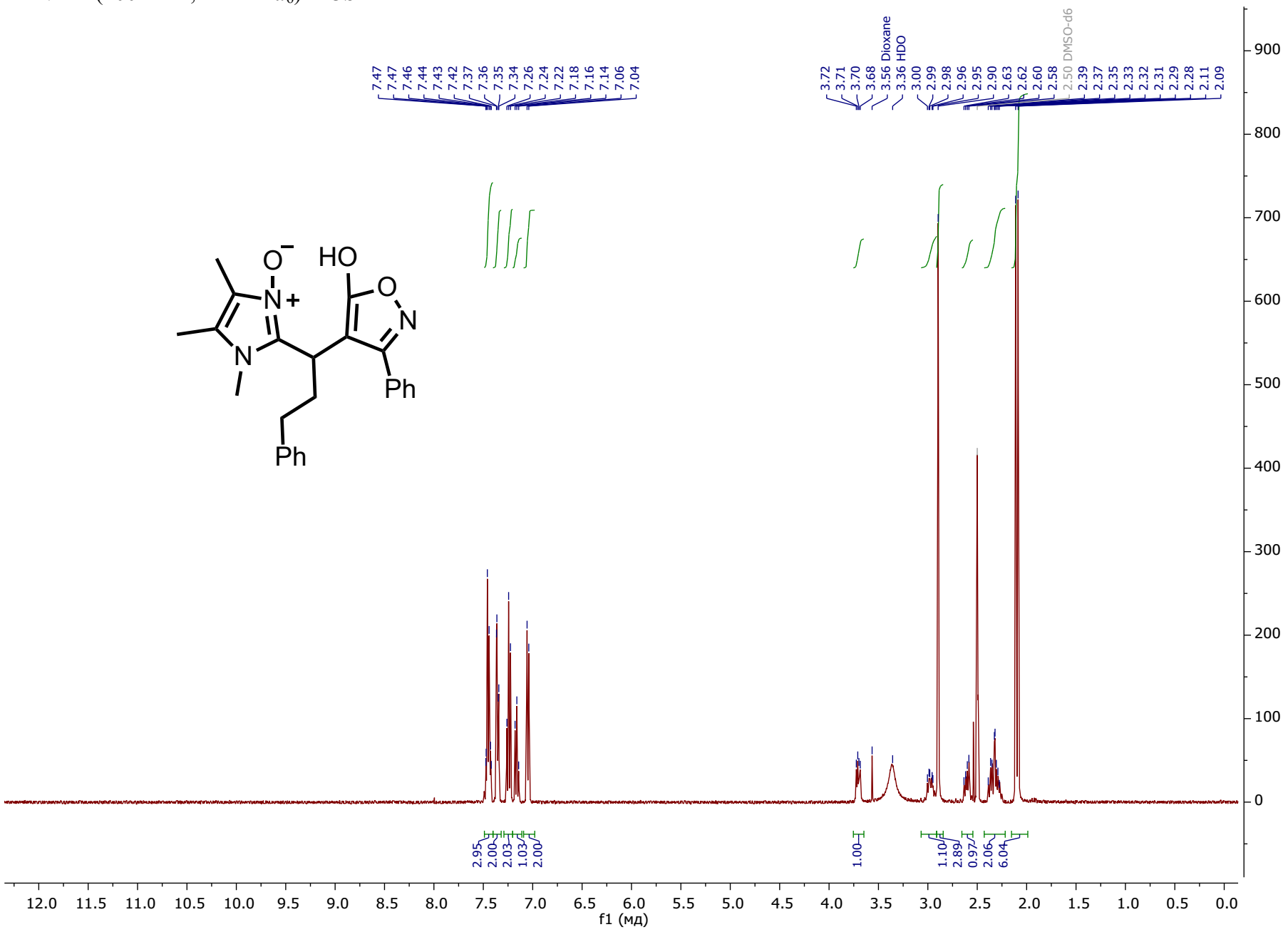
^{1}H NMR (400 MHz, DMSO-d_6) of **3a**



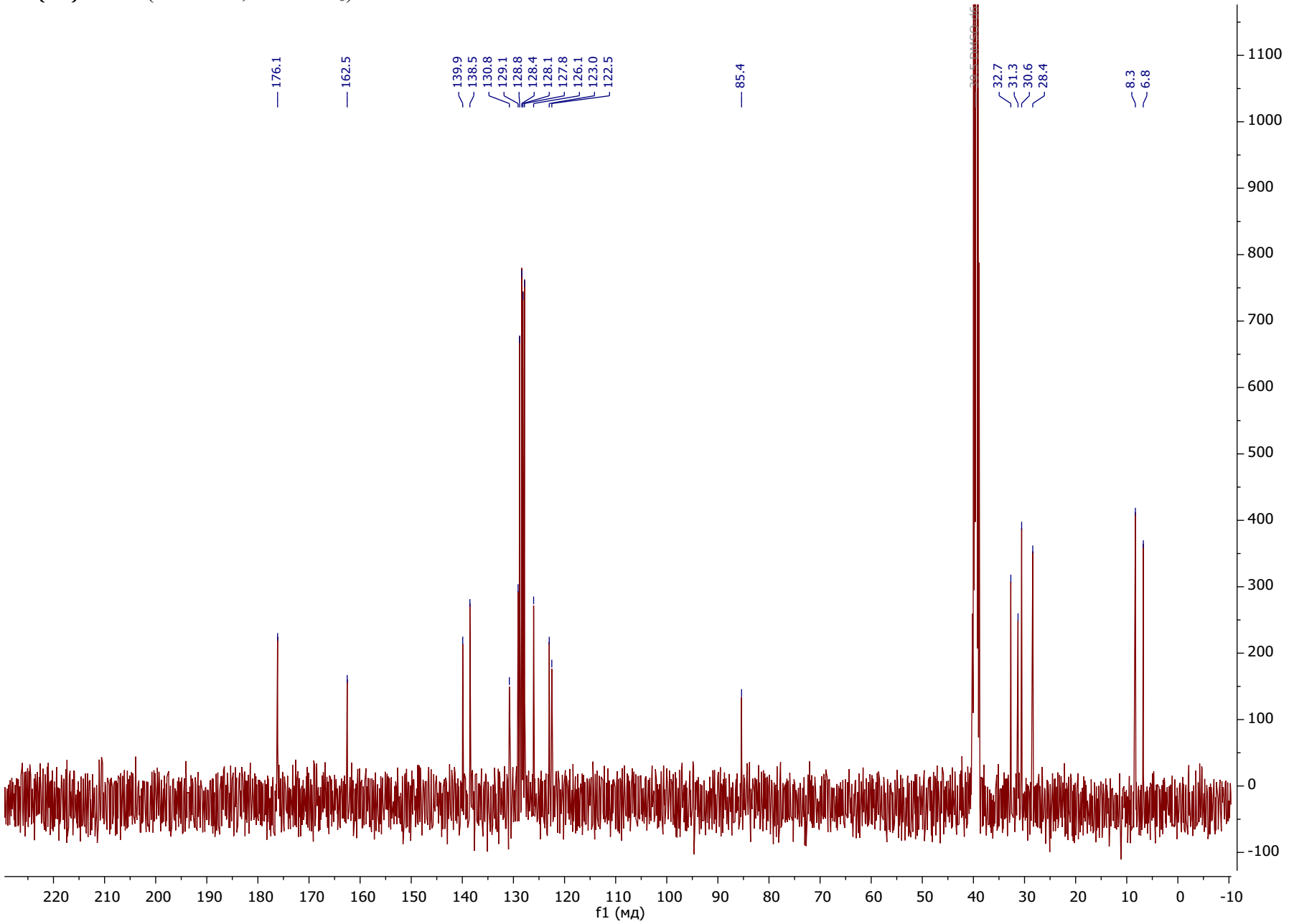
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3a**



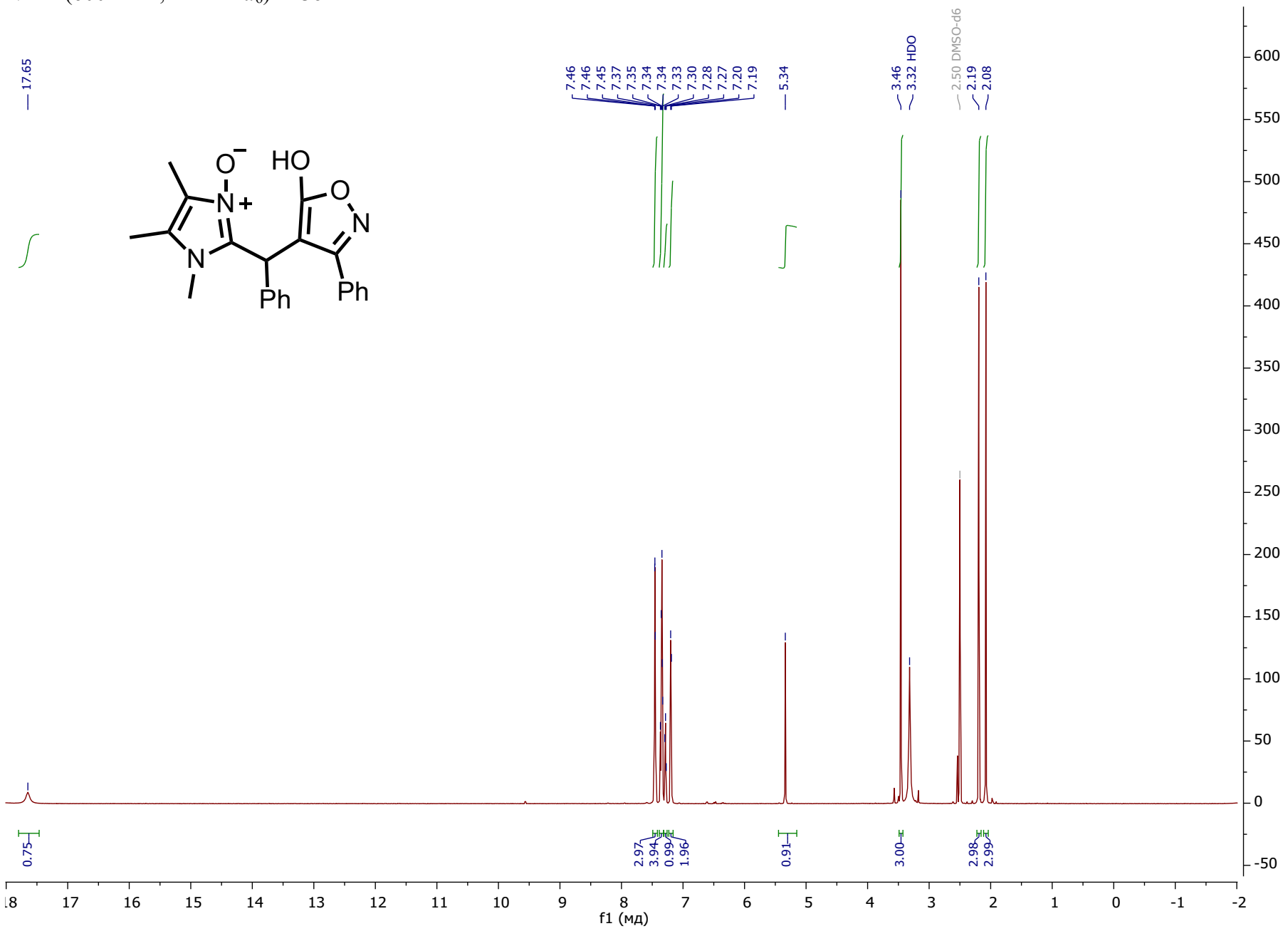
^{1}H NMR (400 MHz, DMSO-d_6) of **3b**



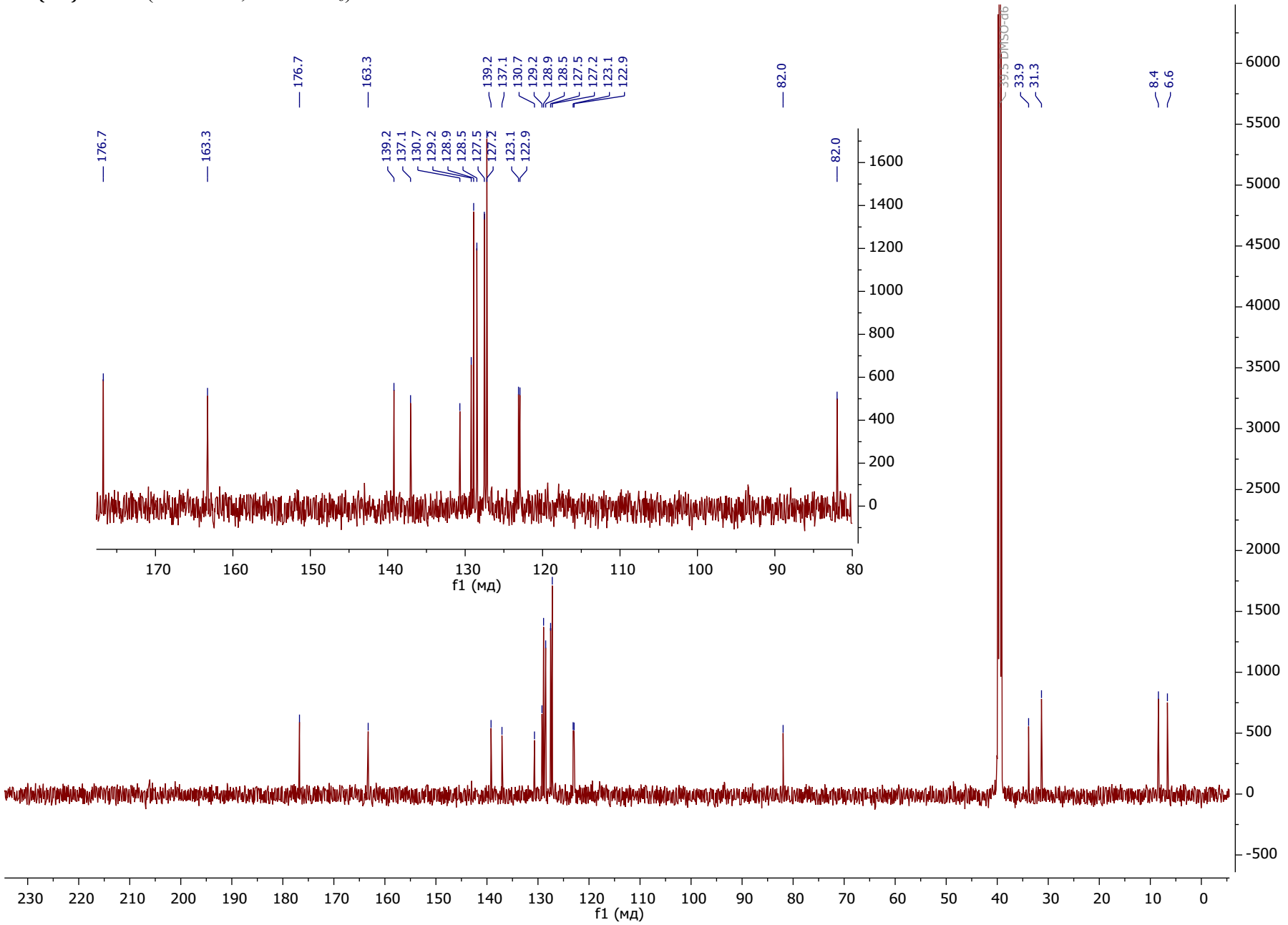
$^{13}C\{^{1}H\}$ NMR (101 MHz, DMSO-d_6) of **3b**



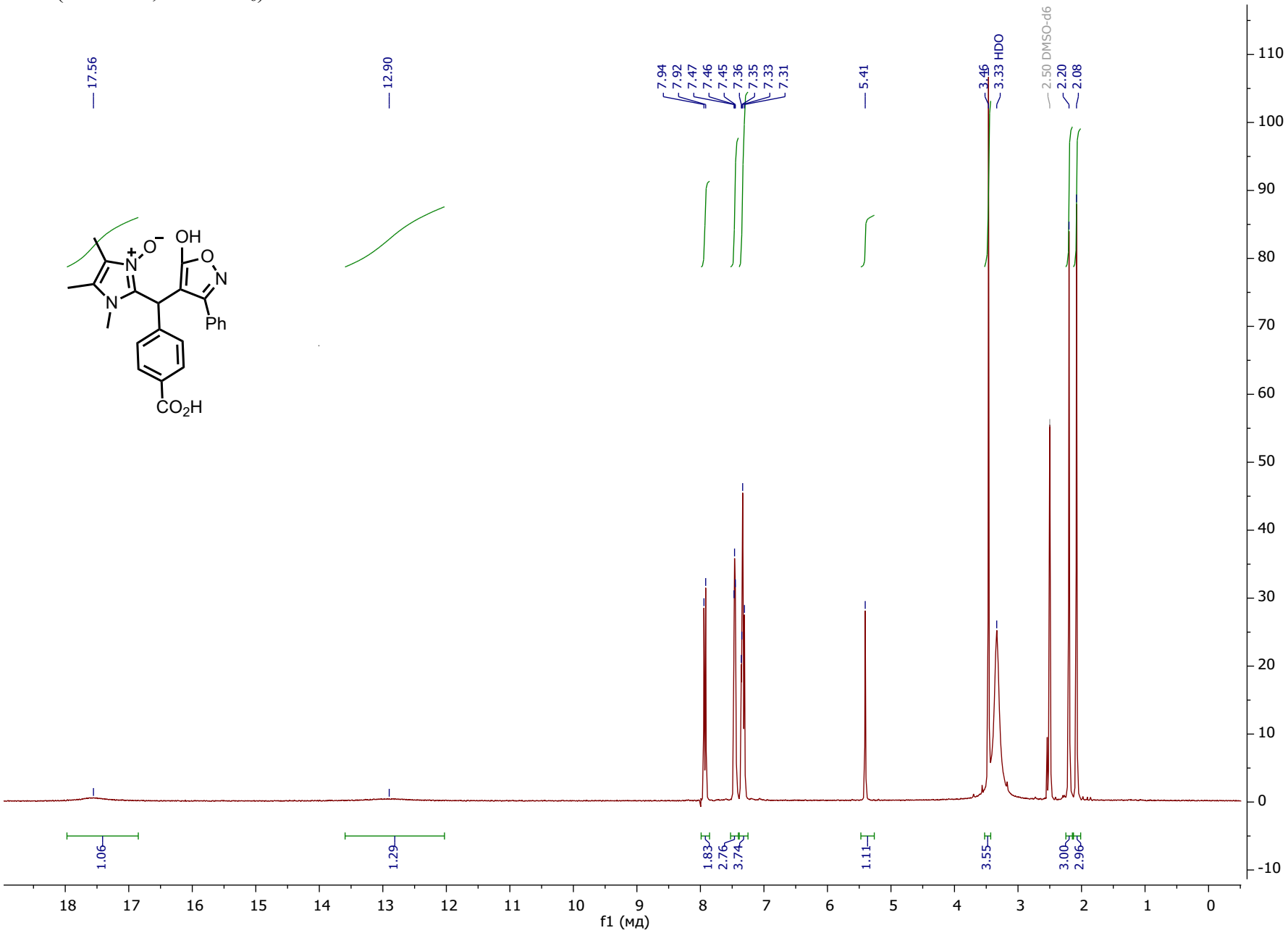
^{1}H NMR (600 MHz, DMSO-d_6) of **3c**



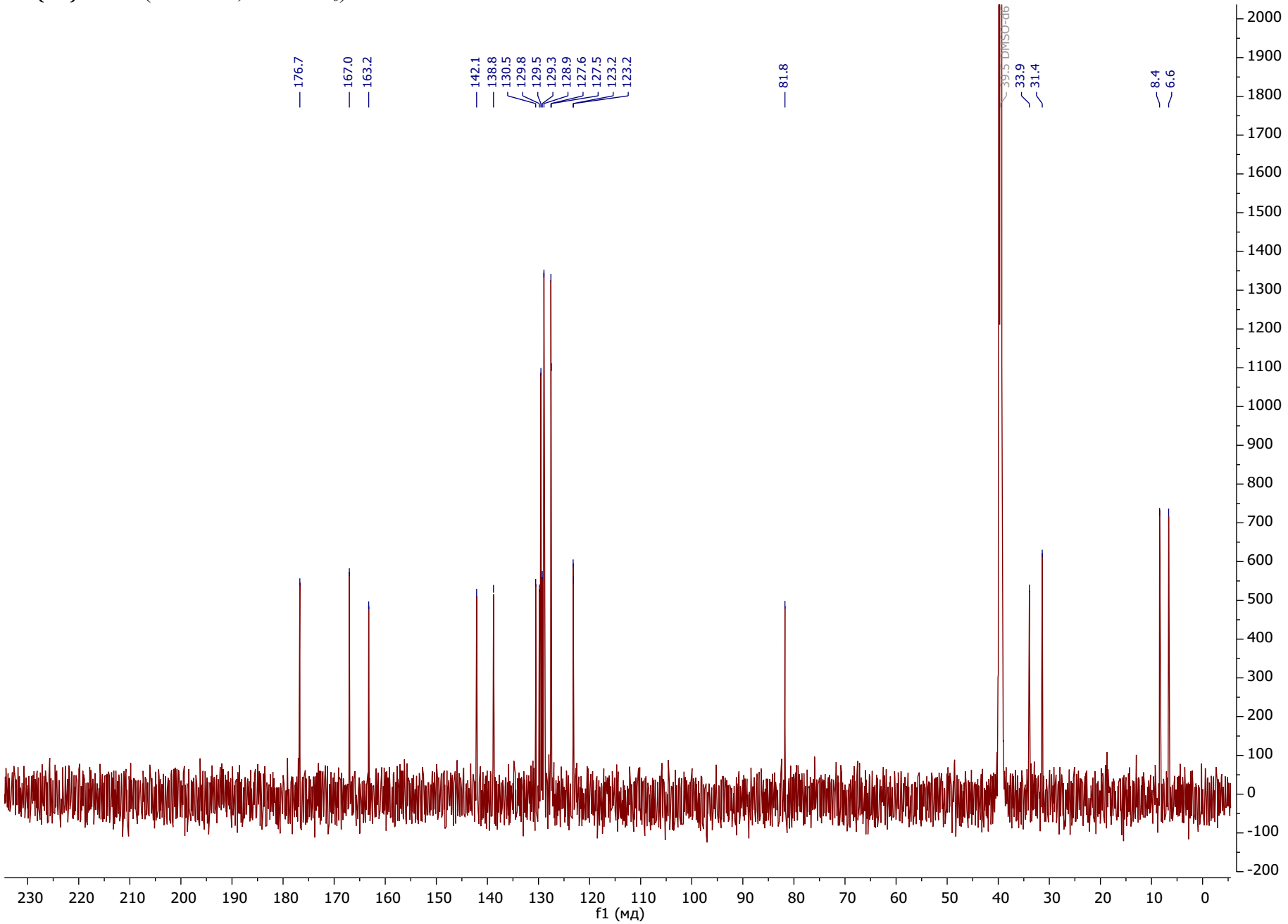
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3c**



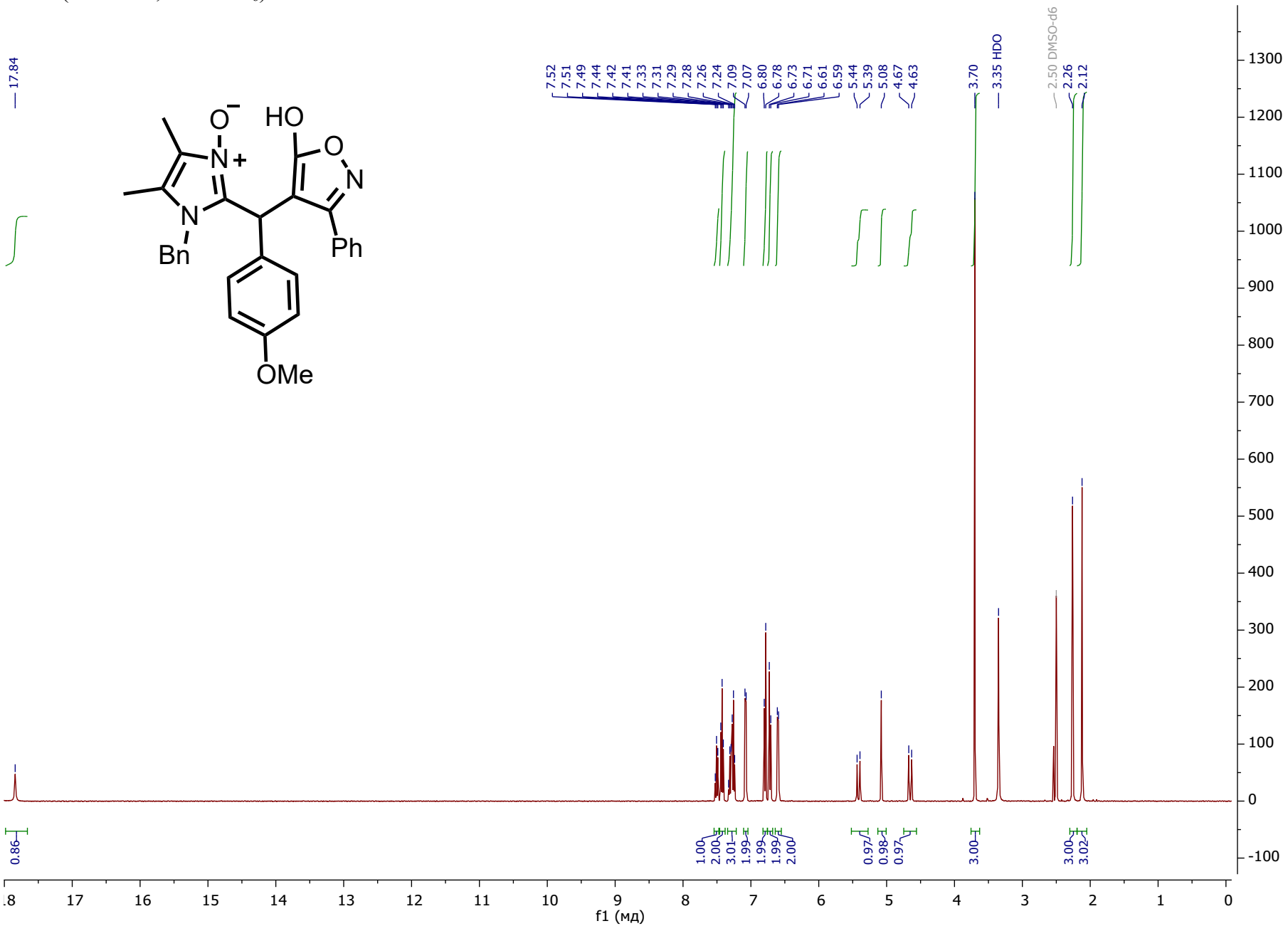
^{1}H NMR (300 MHz, DMSO-d_6) of **3d**



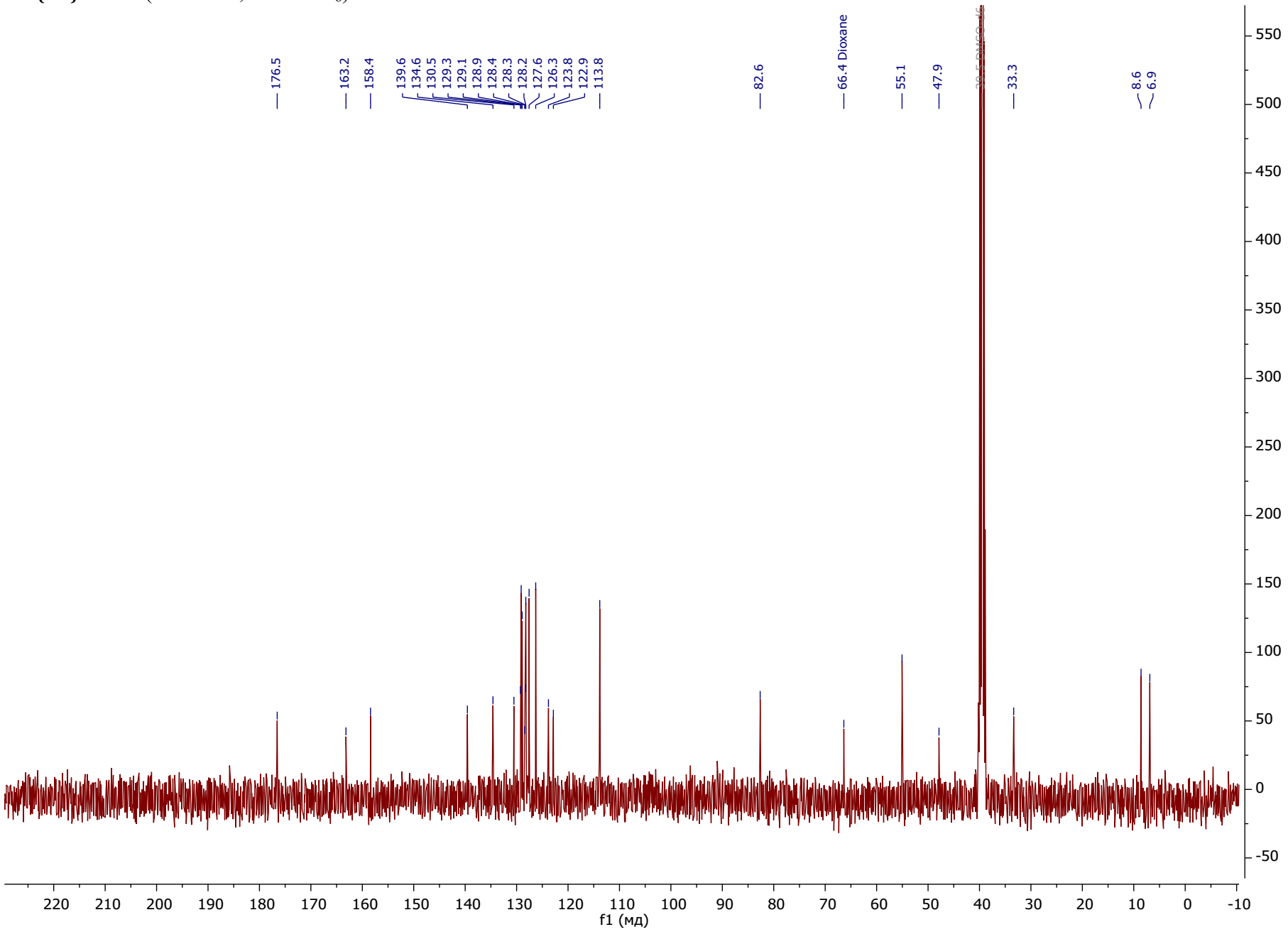
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3d**



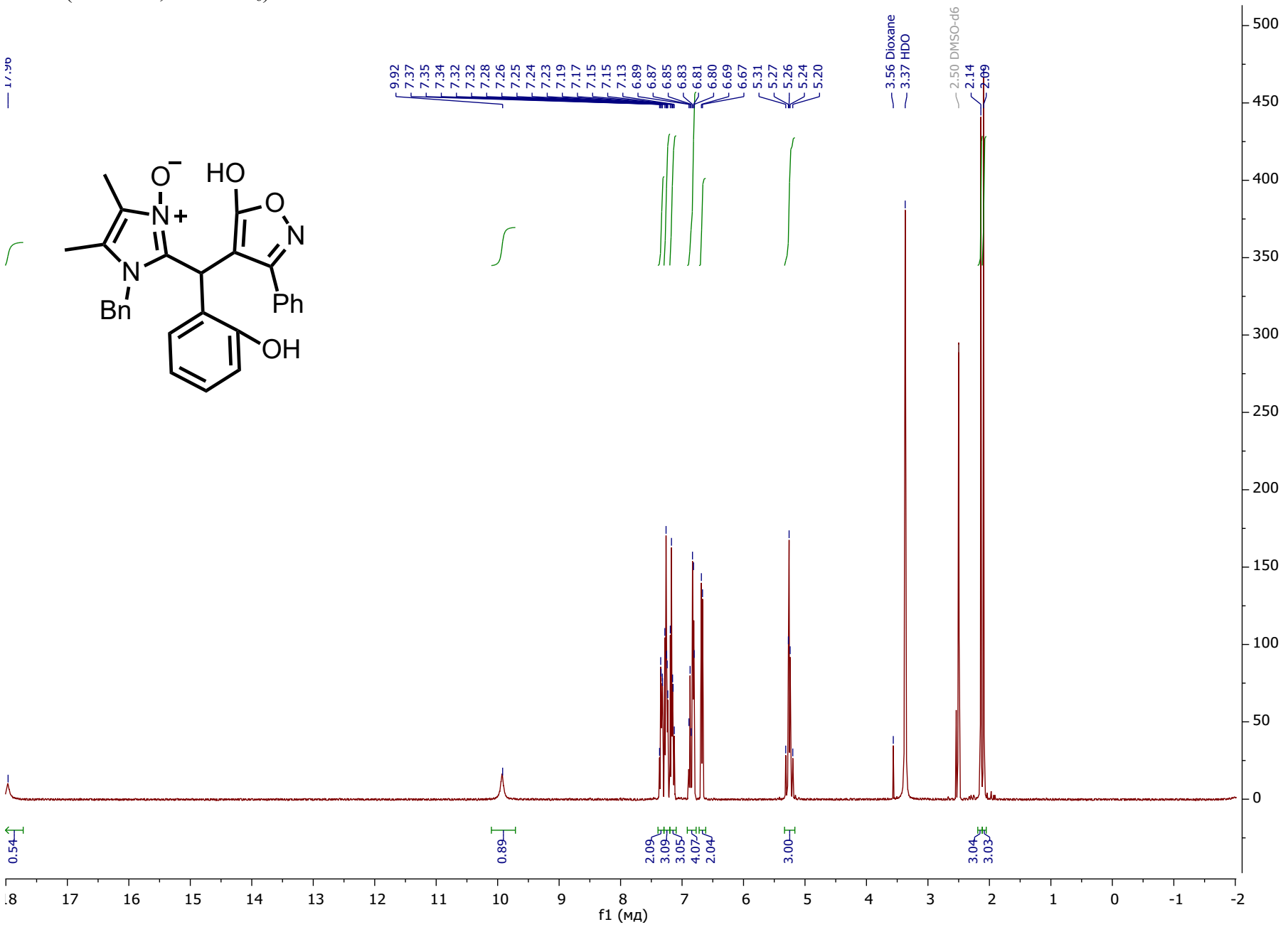
^{1}H NMR (400 MHz, DMSO-d_6) of **3e**



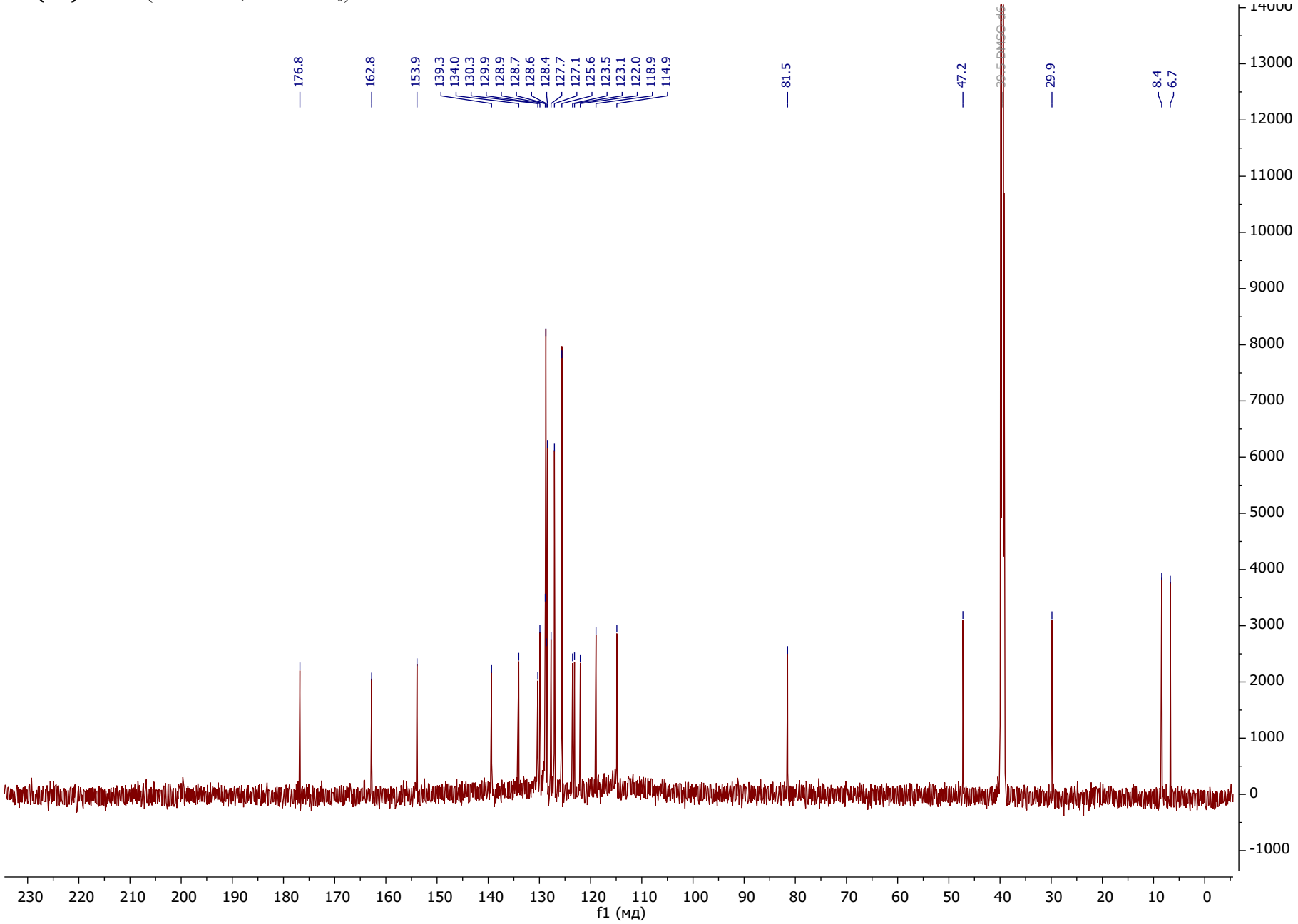
$^{13}C\{^{1}H\}$ NMR (101 MHz, DMSO-d_6) of **3e**



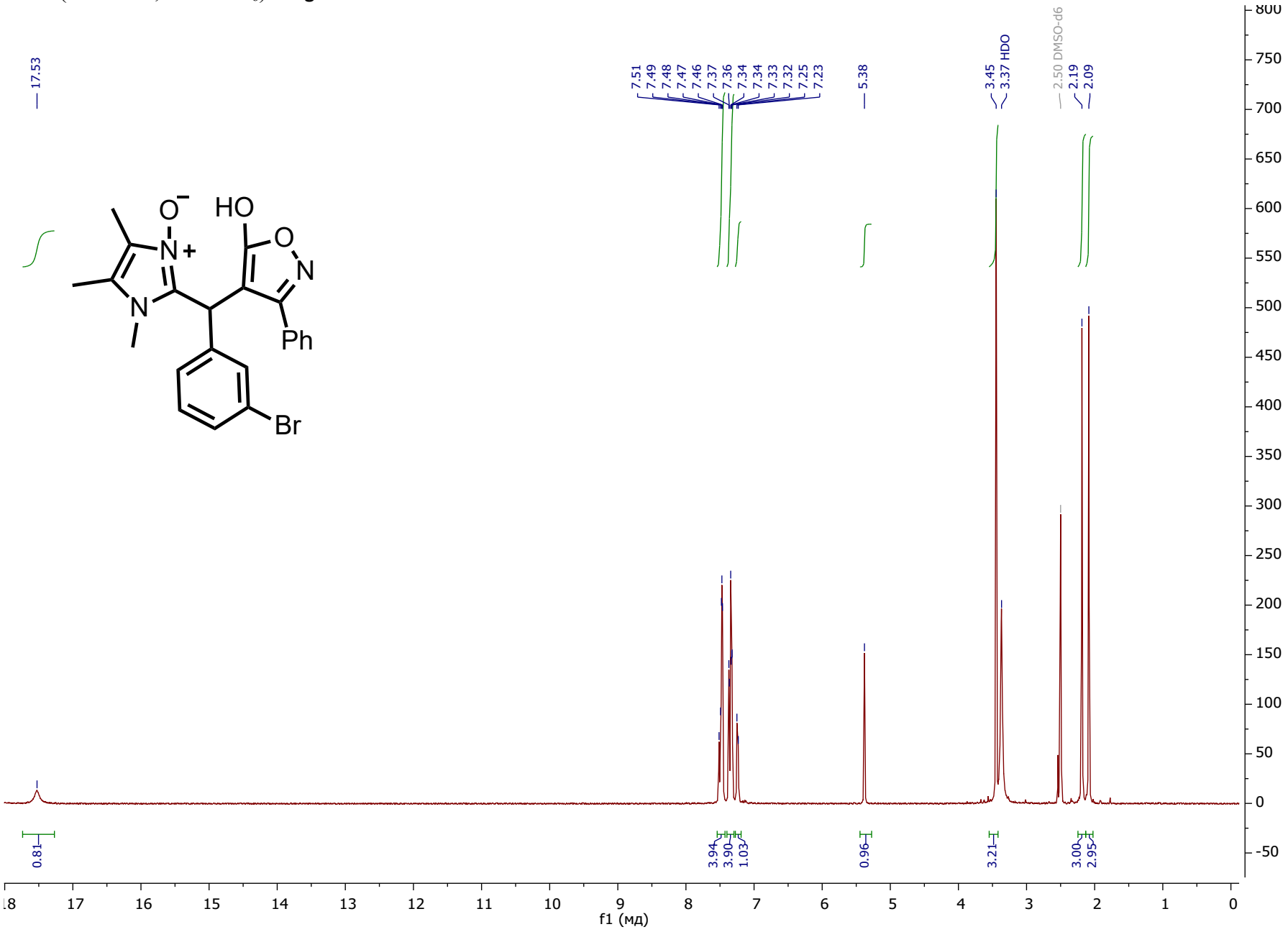
^{1}H NMR (400 MHz, DMSO-d_6) of **3f**



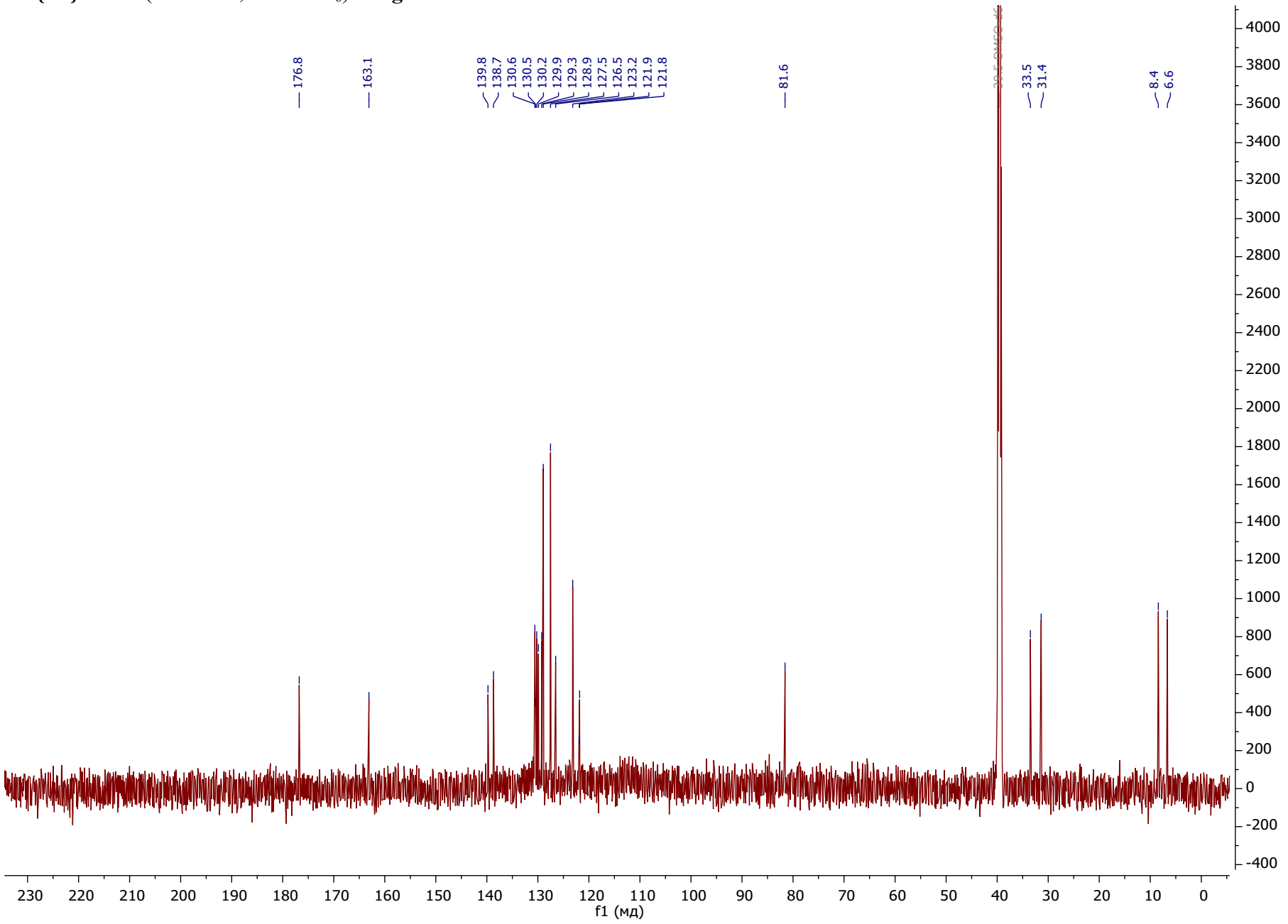
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3f**



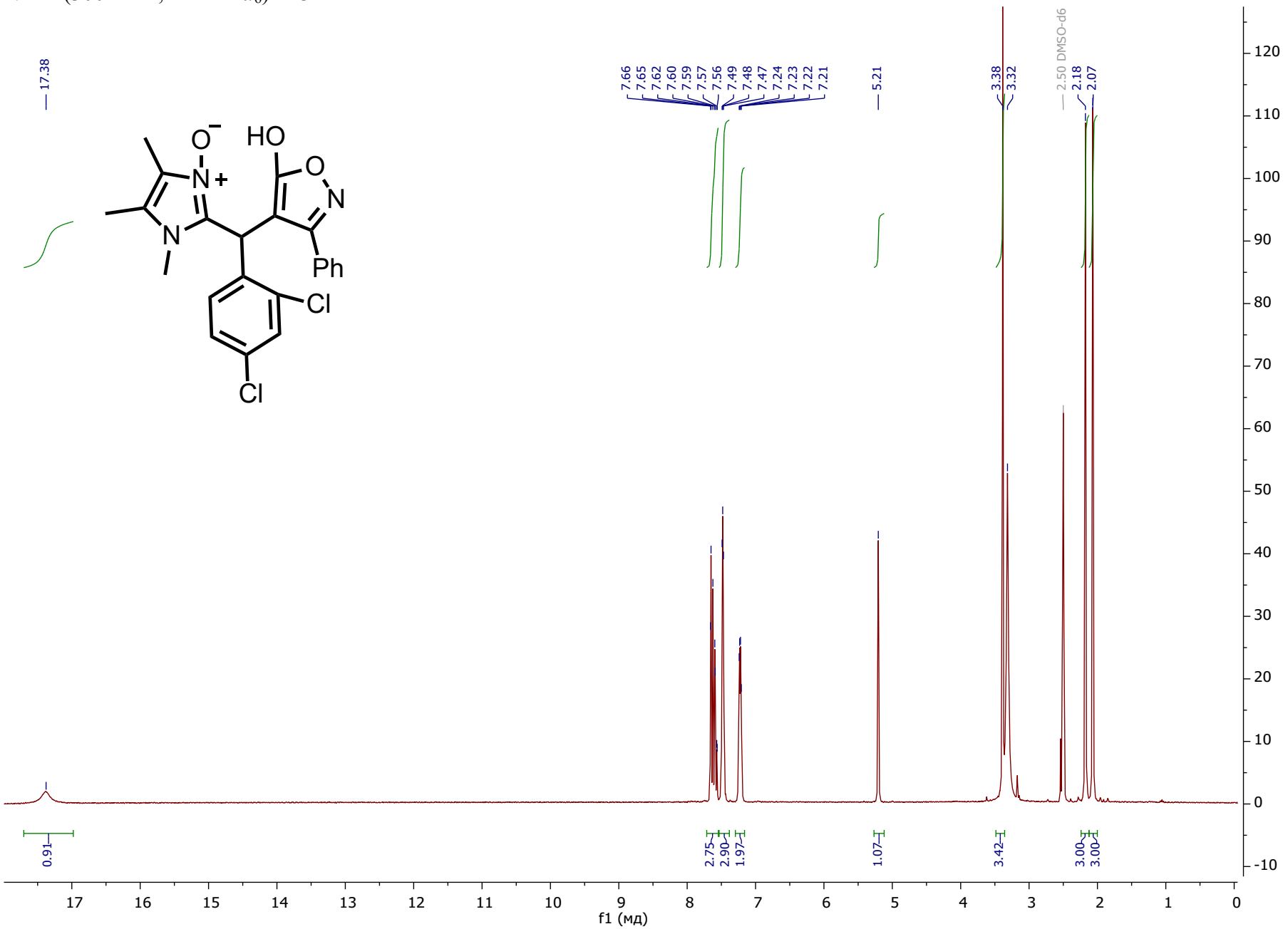
^{1}H NMR (400 MHz, DMSO-d_6) of **3g**



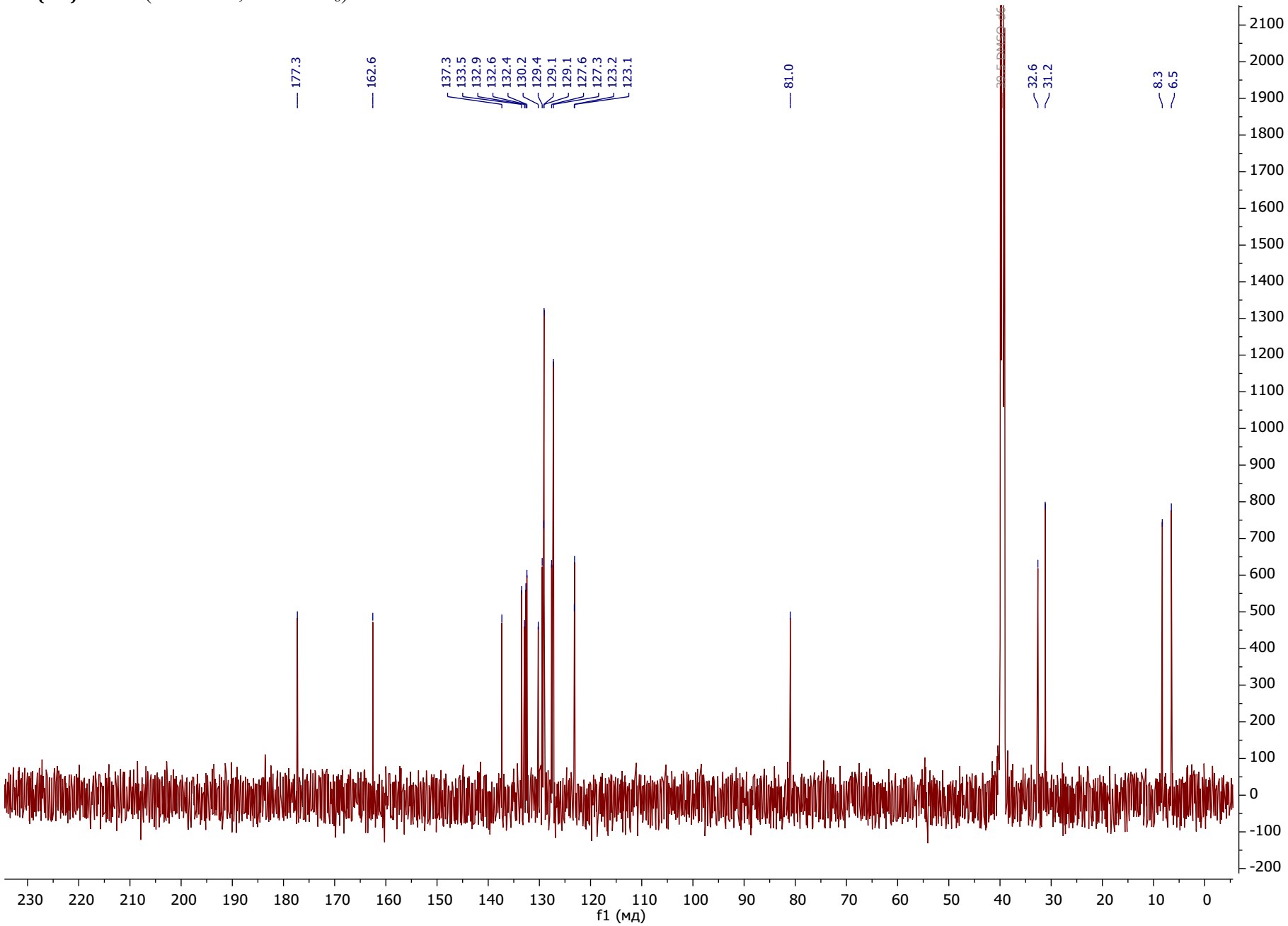
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3g**



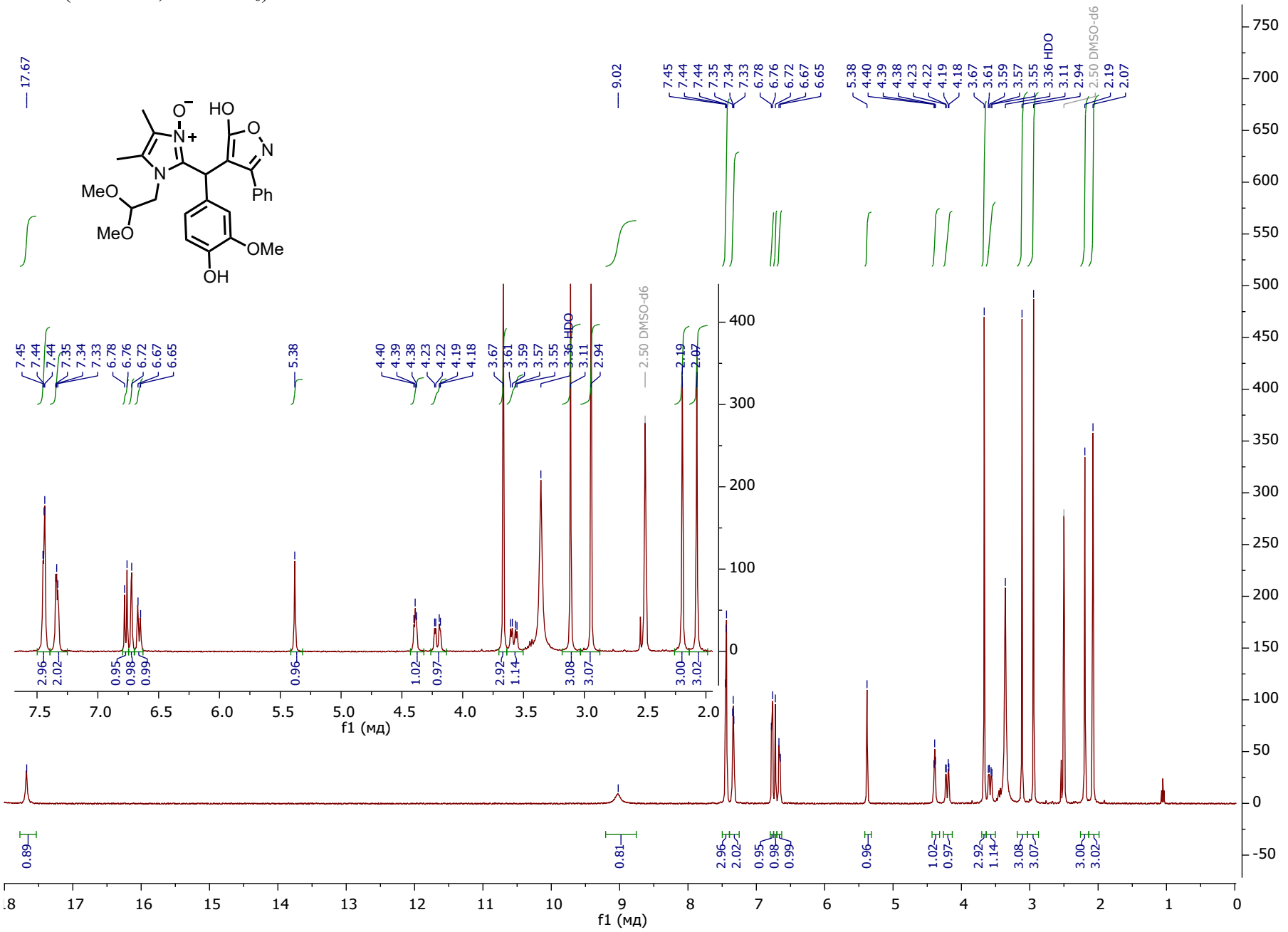
^{1}H NMR (300 MHz, DMSO-d_6) of **3h**



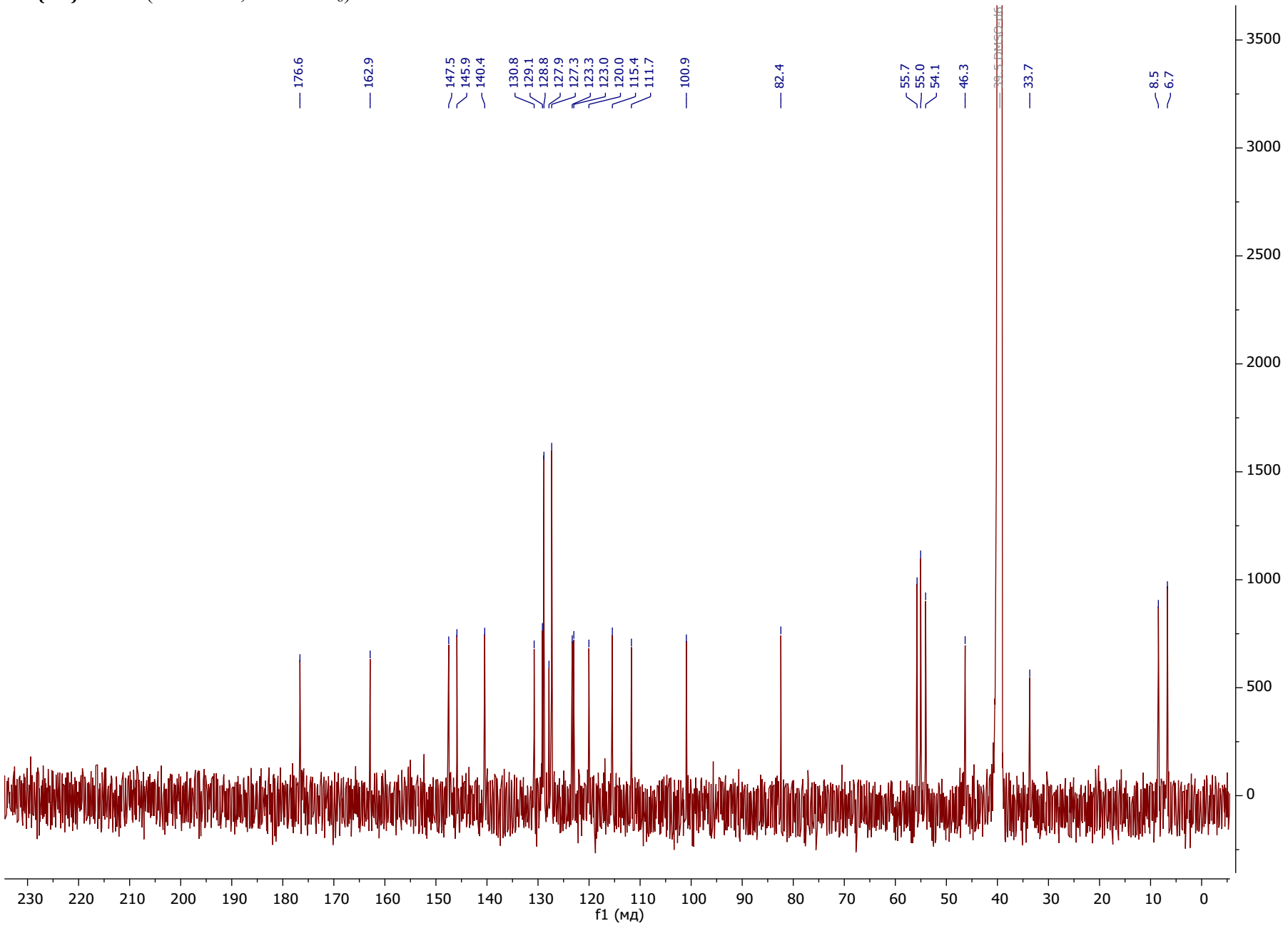
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3h**



^{1}H NMR (400 MHz, DMSO-d_6) of **3i**



$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3i**



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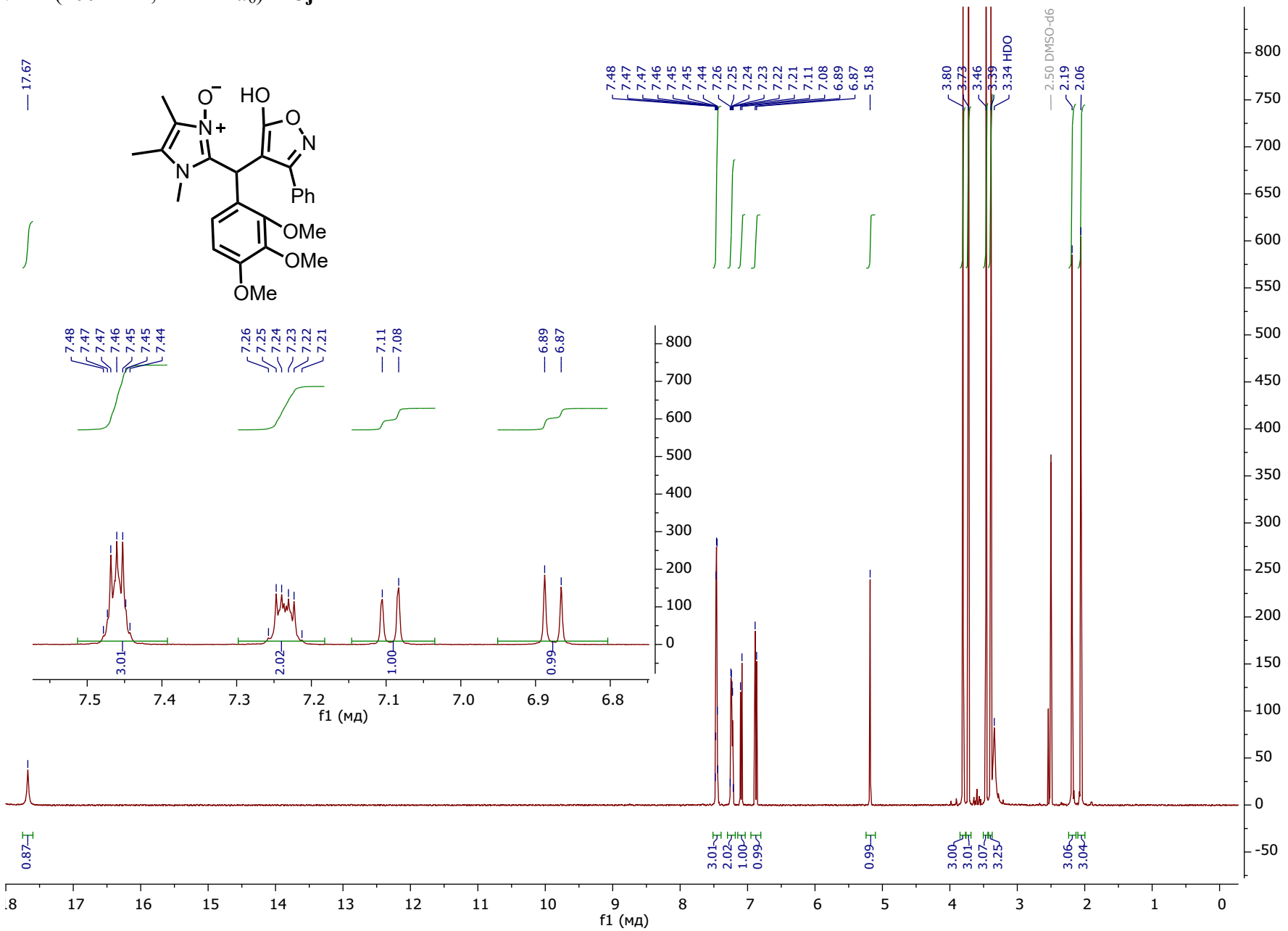
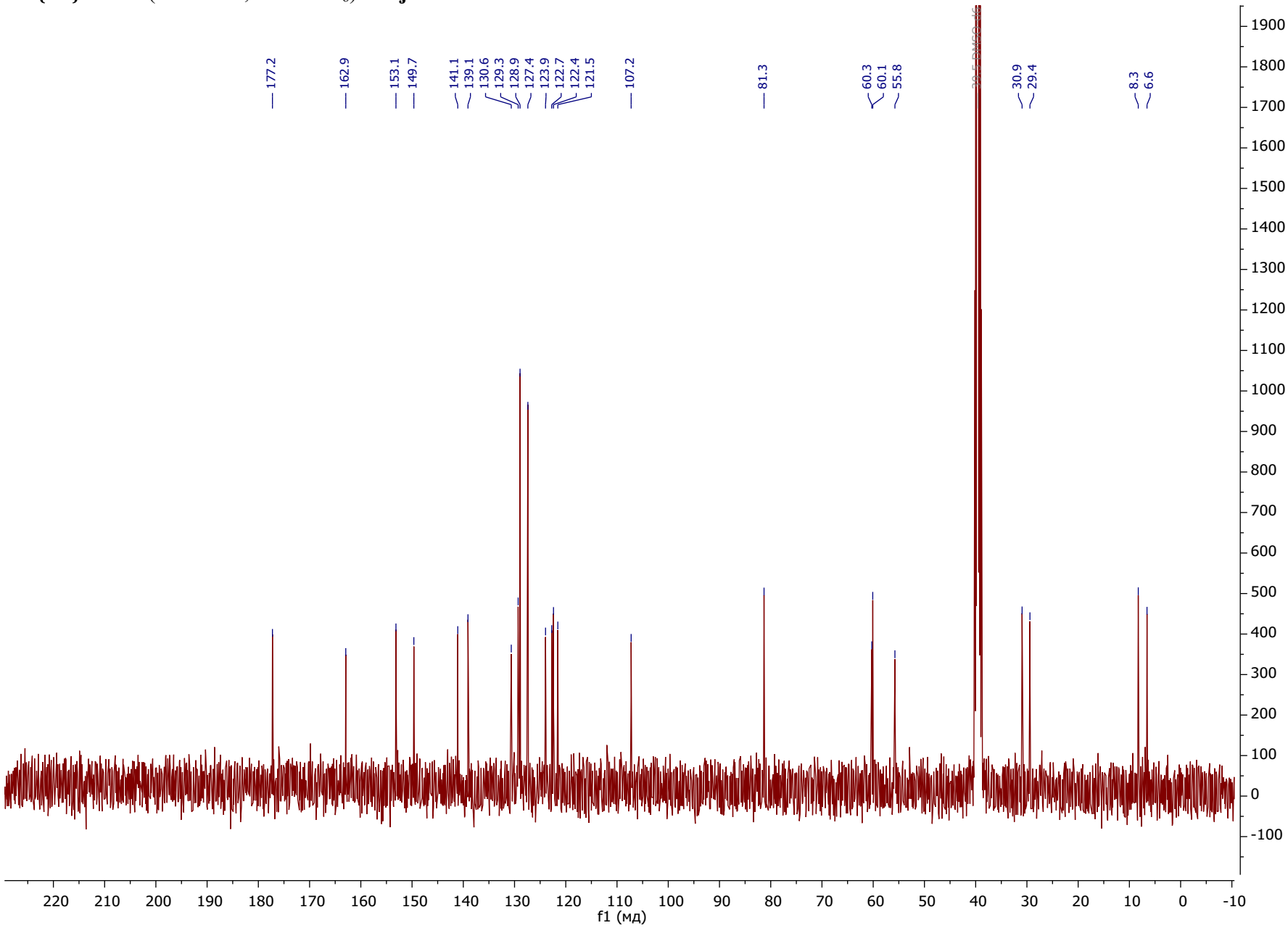
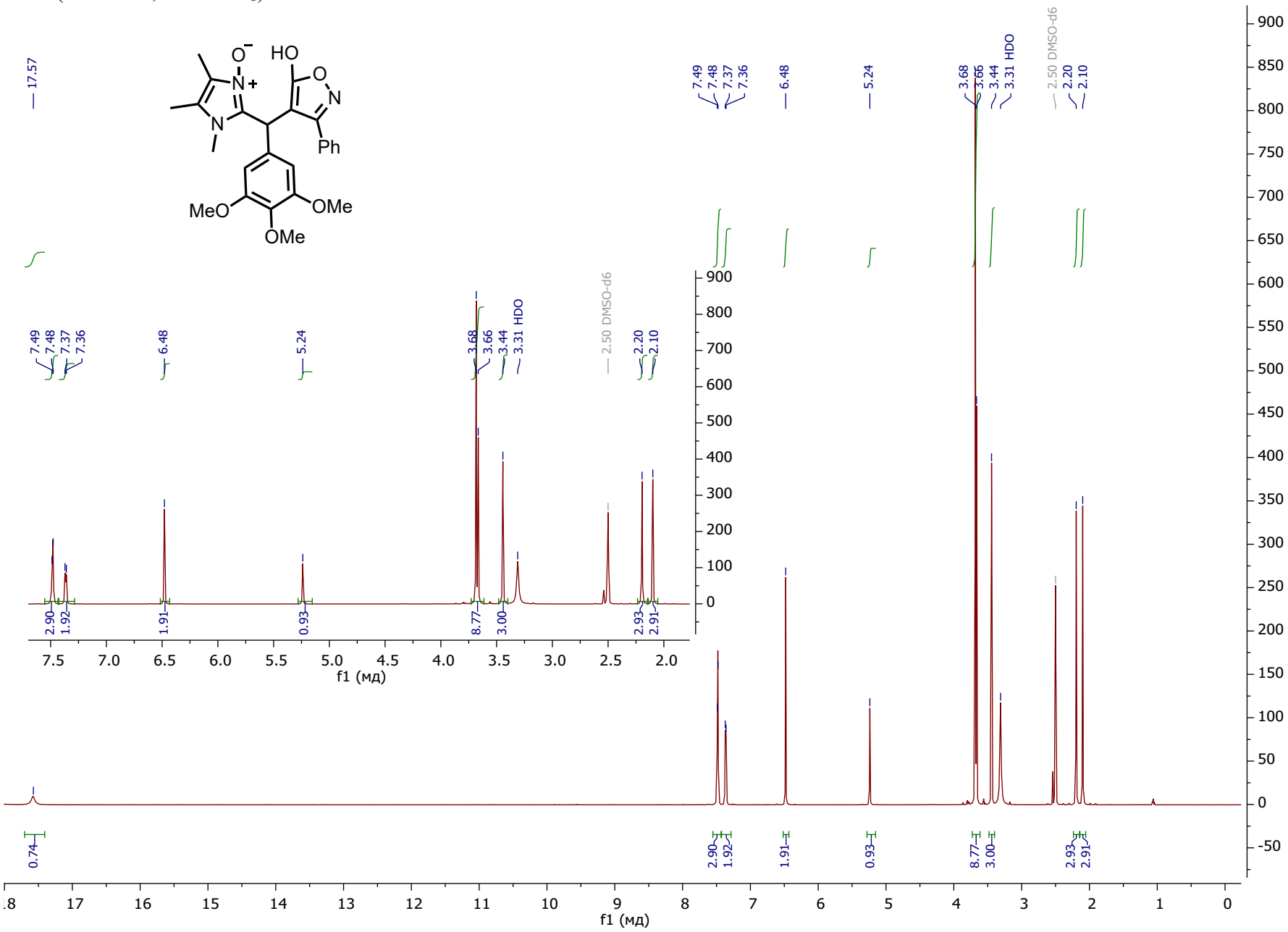


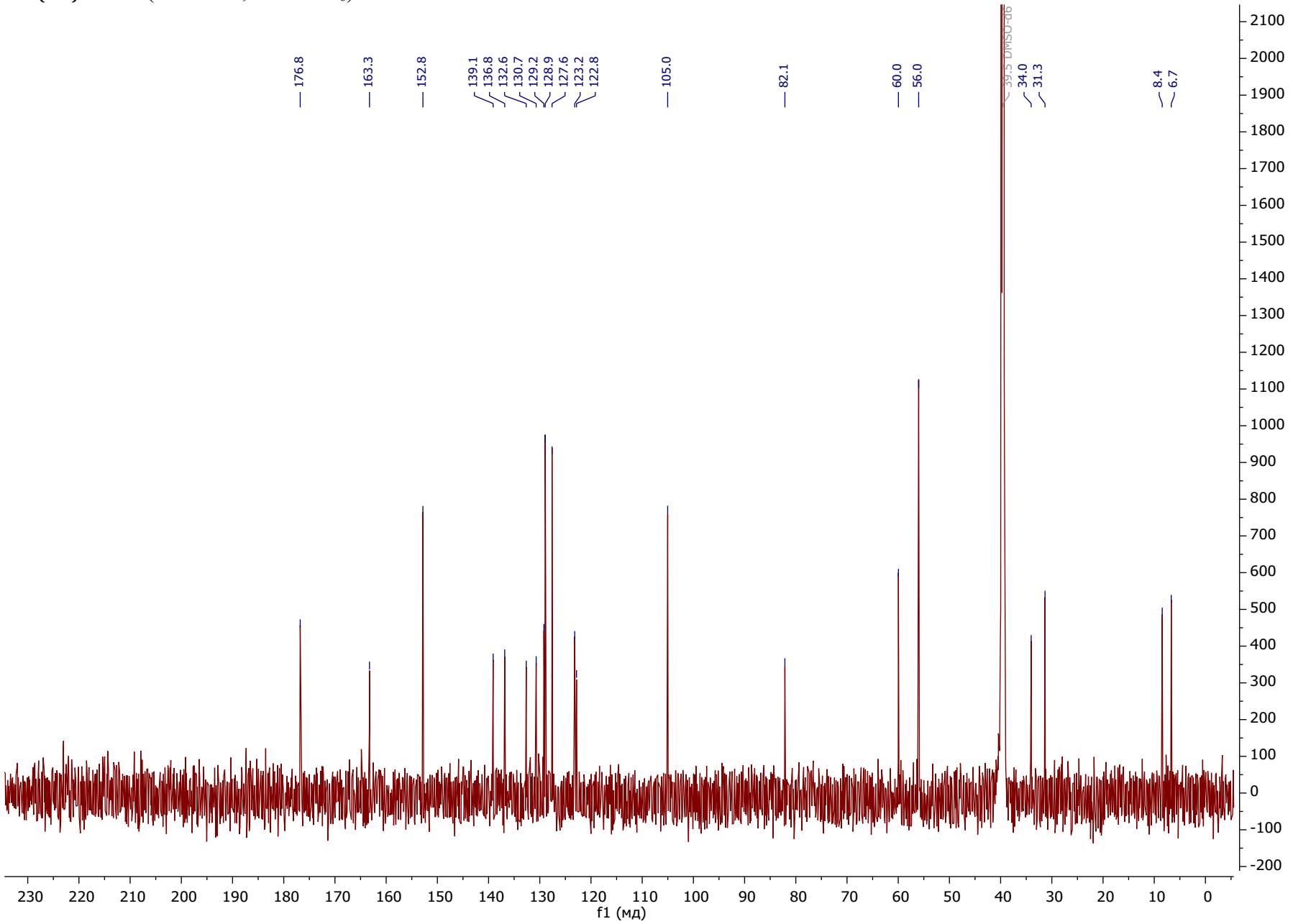
$^{13}C\{^{1}H\}$ NMR (101 MHz, DMSO-d_6) of **3j**



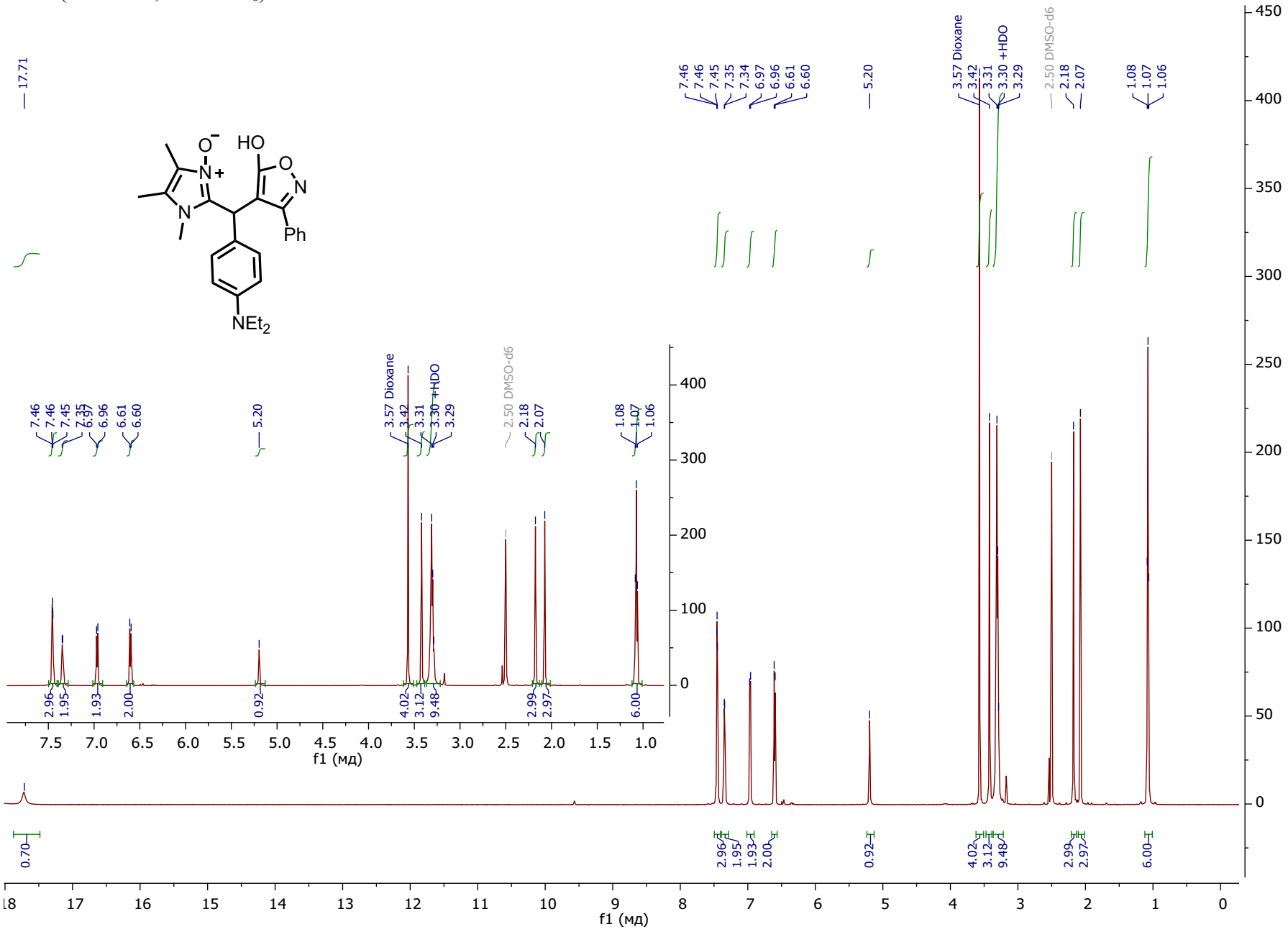
^{1}H NMR (600 MHz, DMSO-d_6) of **3k**



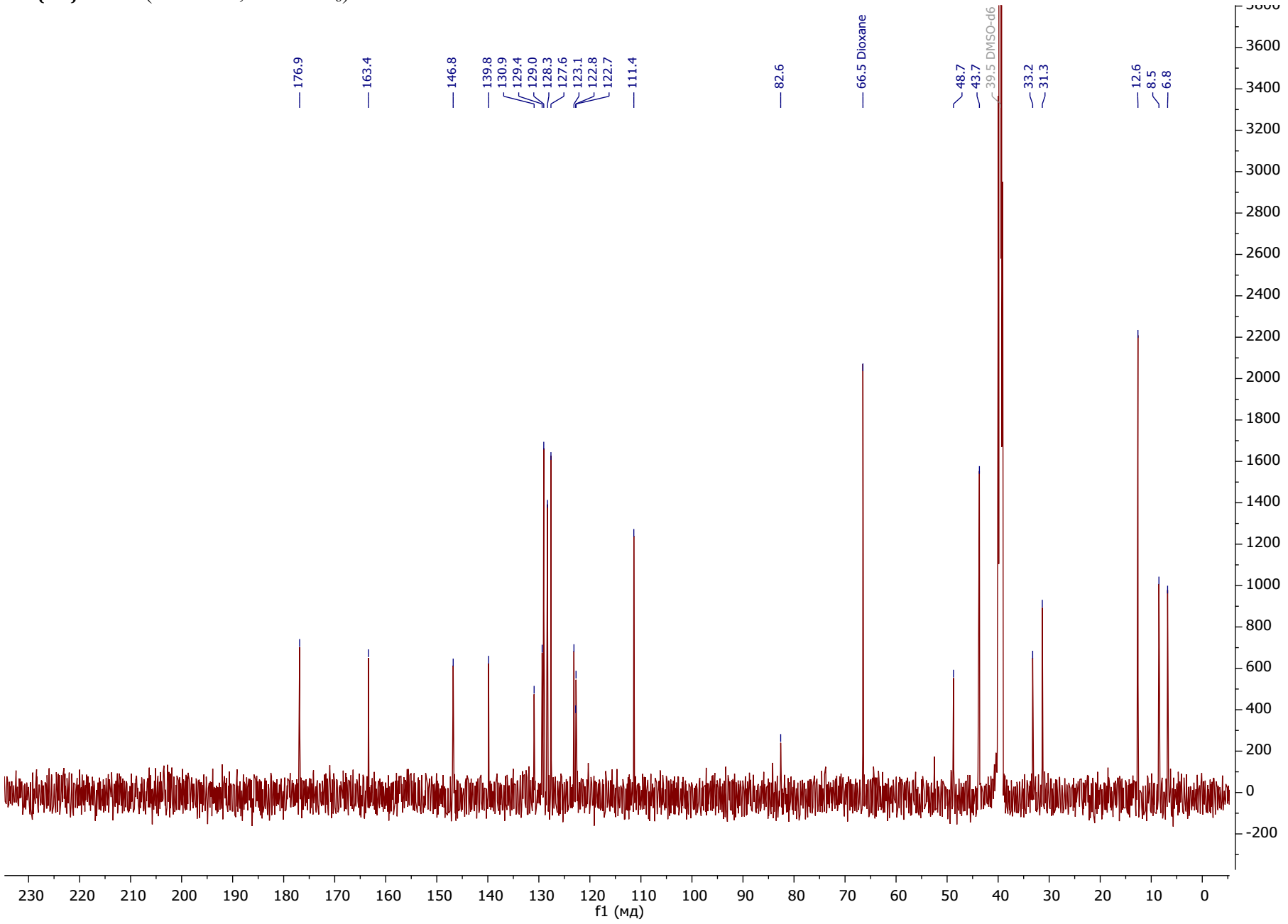
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3k**



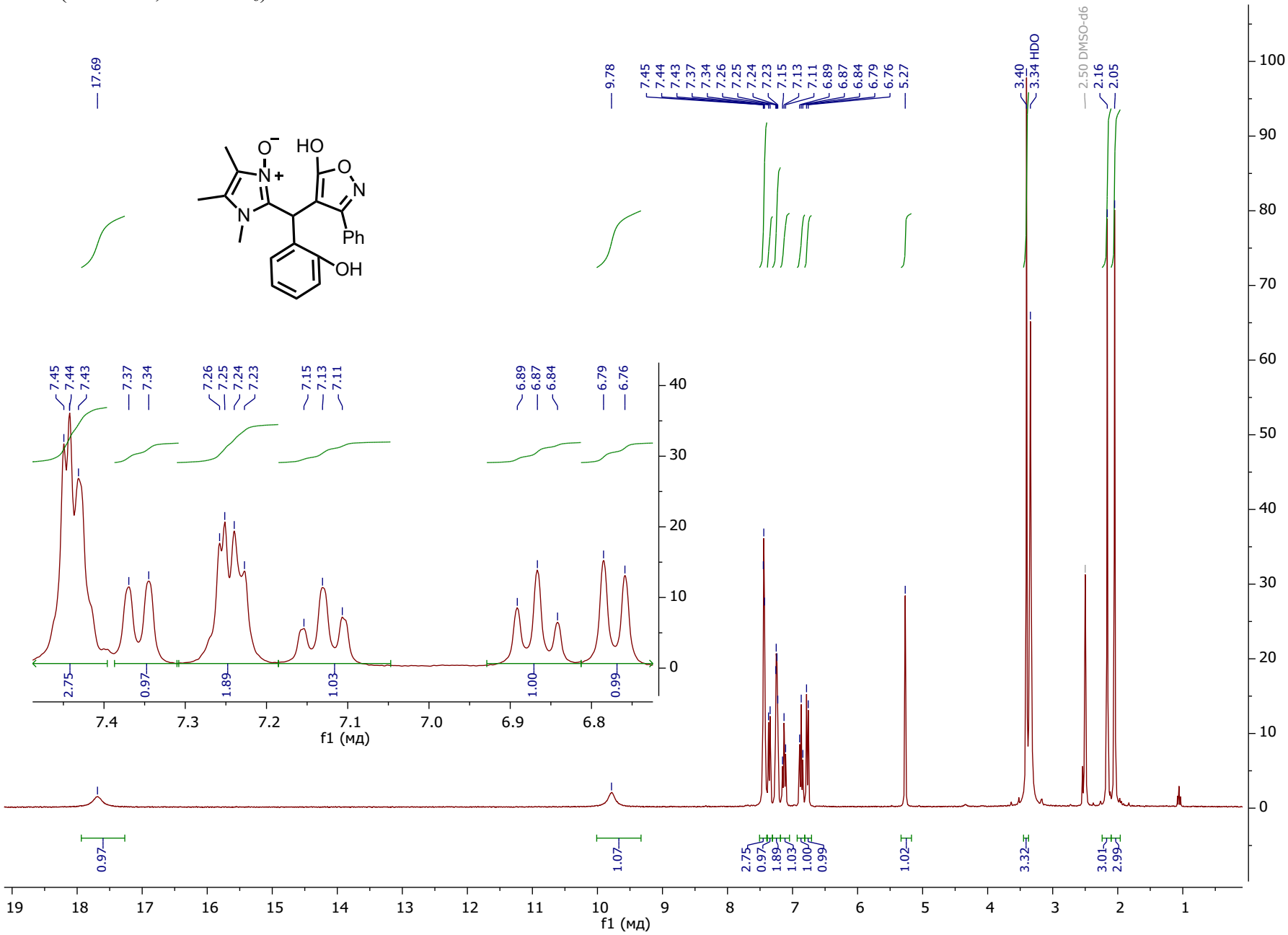
^{1}H NMR (600 MHz, DMSO-d_6) of **3l**



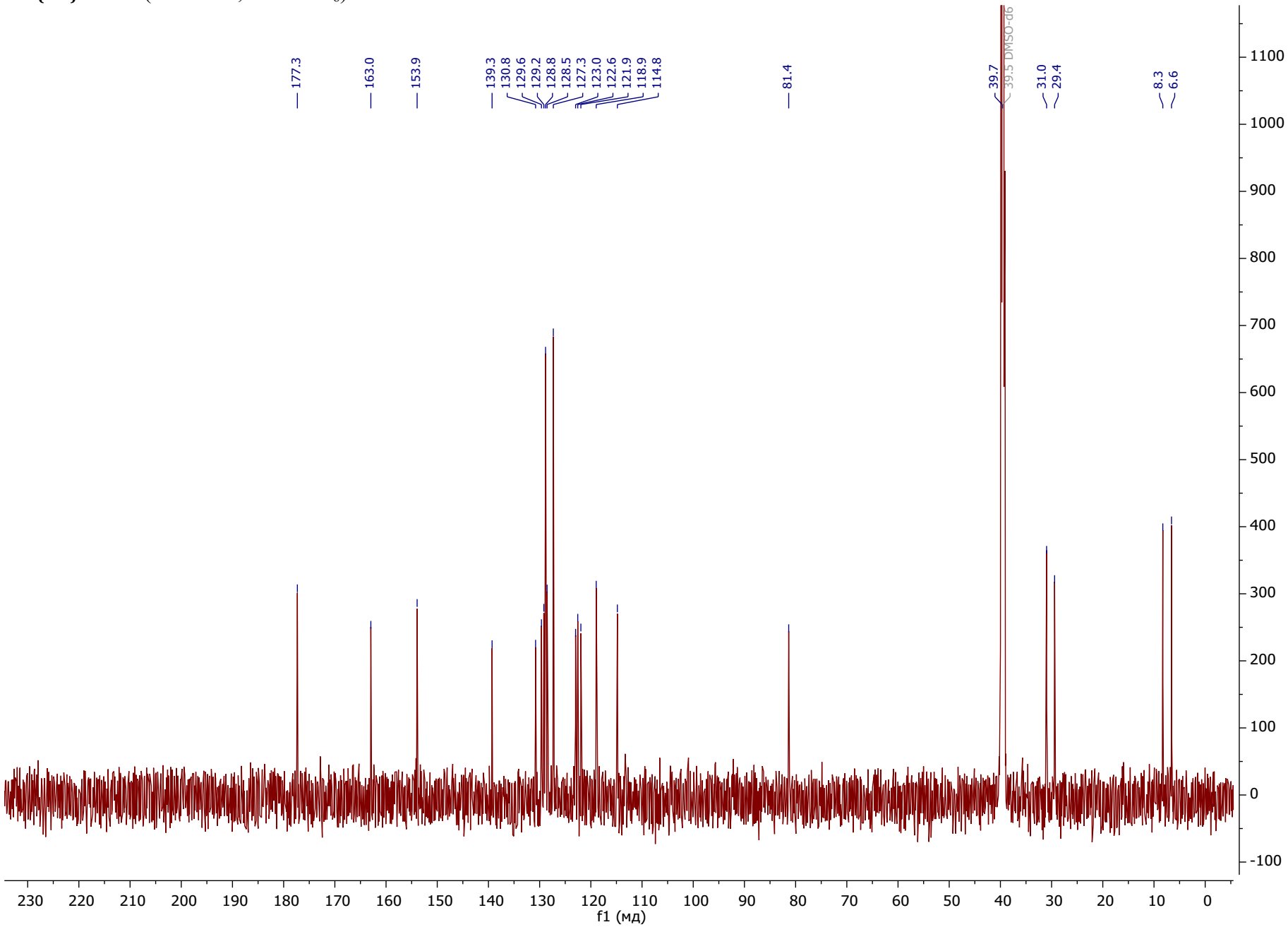
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3l**



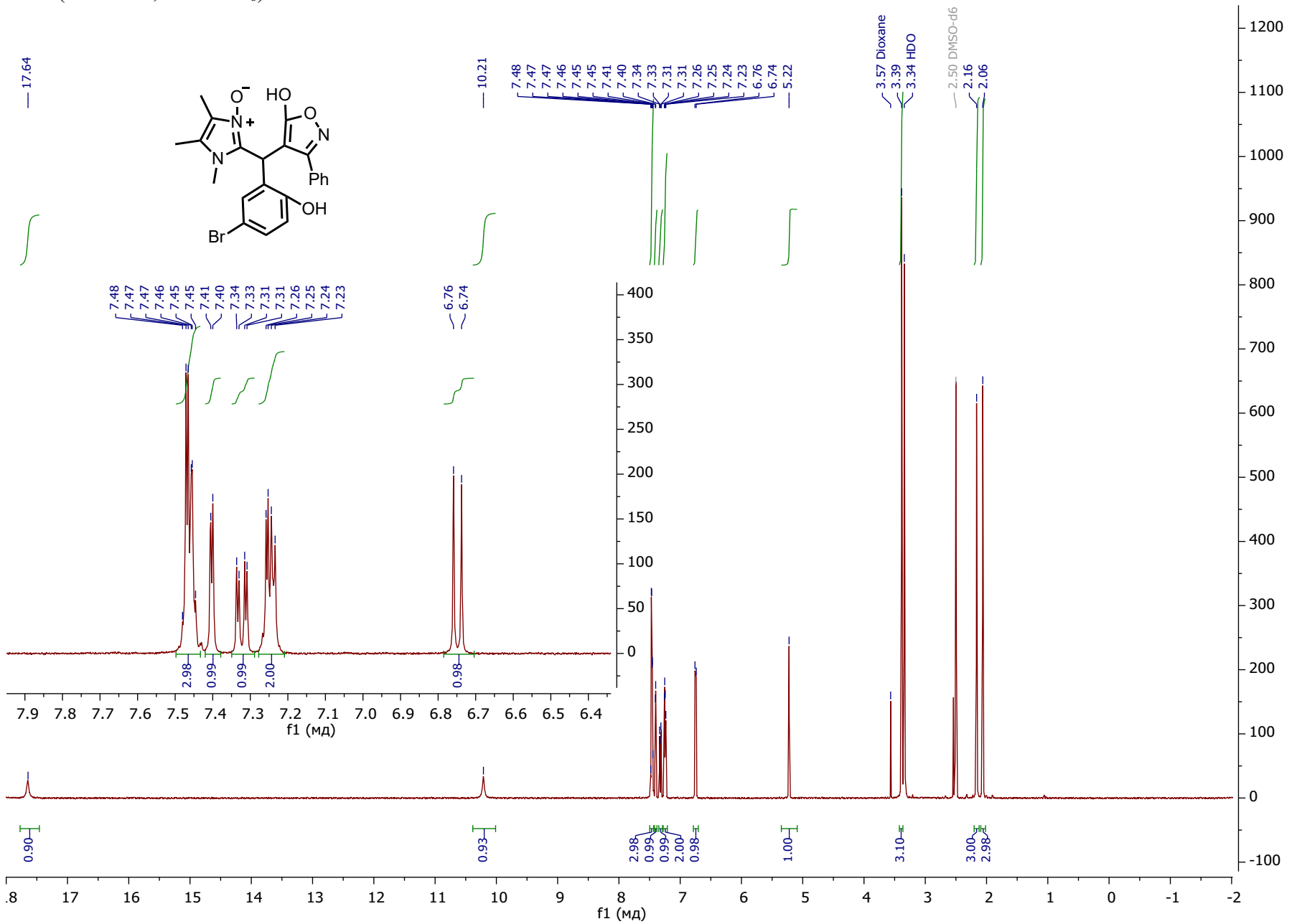
^{1}H NMR (300 MHz, DMSO-d_6) of **3m**



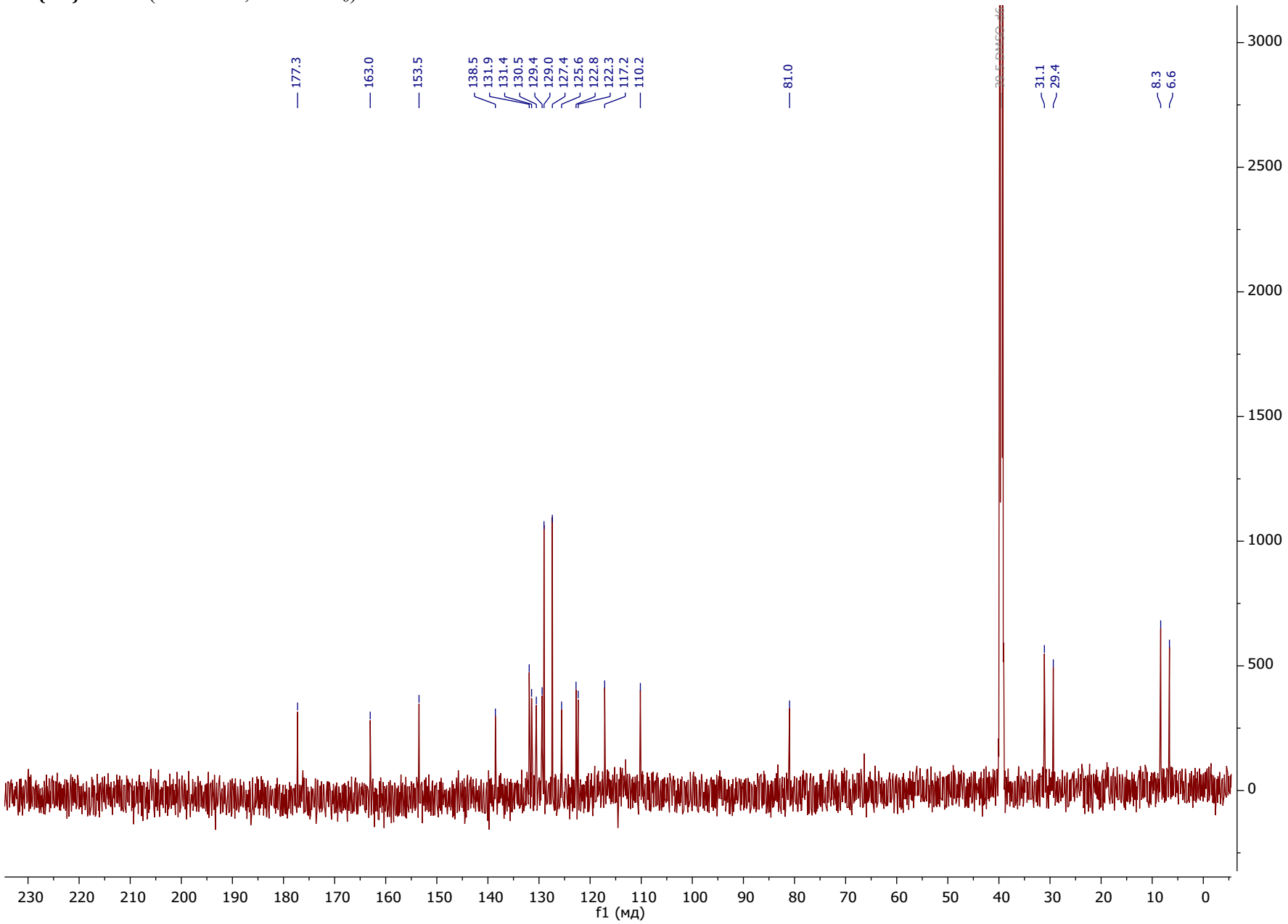
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3m**



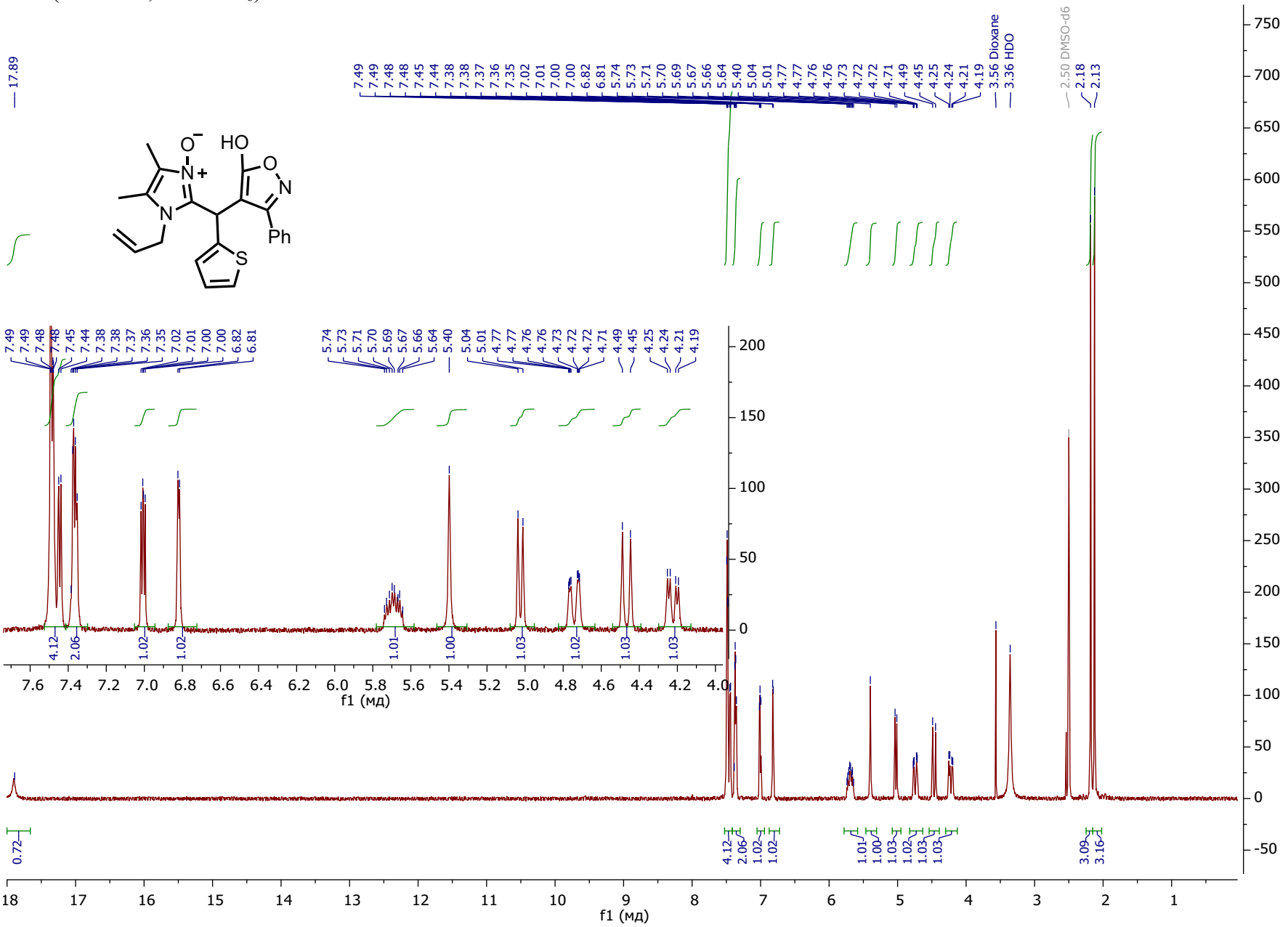
^{1}H NMR (400 MHz, DMSO-d_6) of **3n**



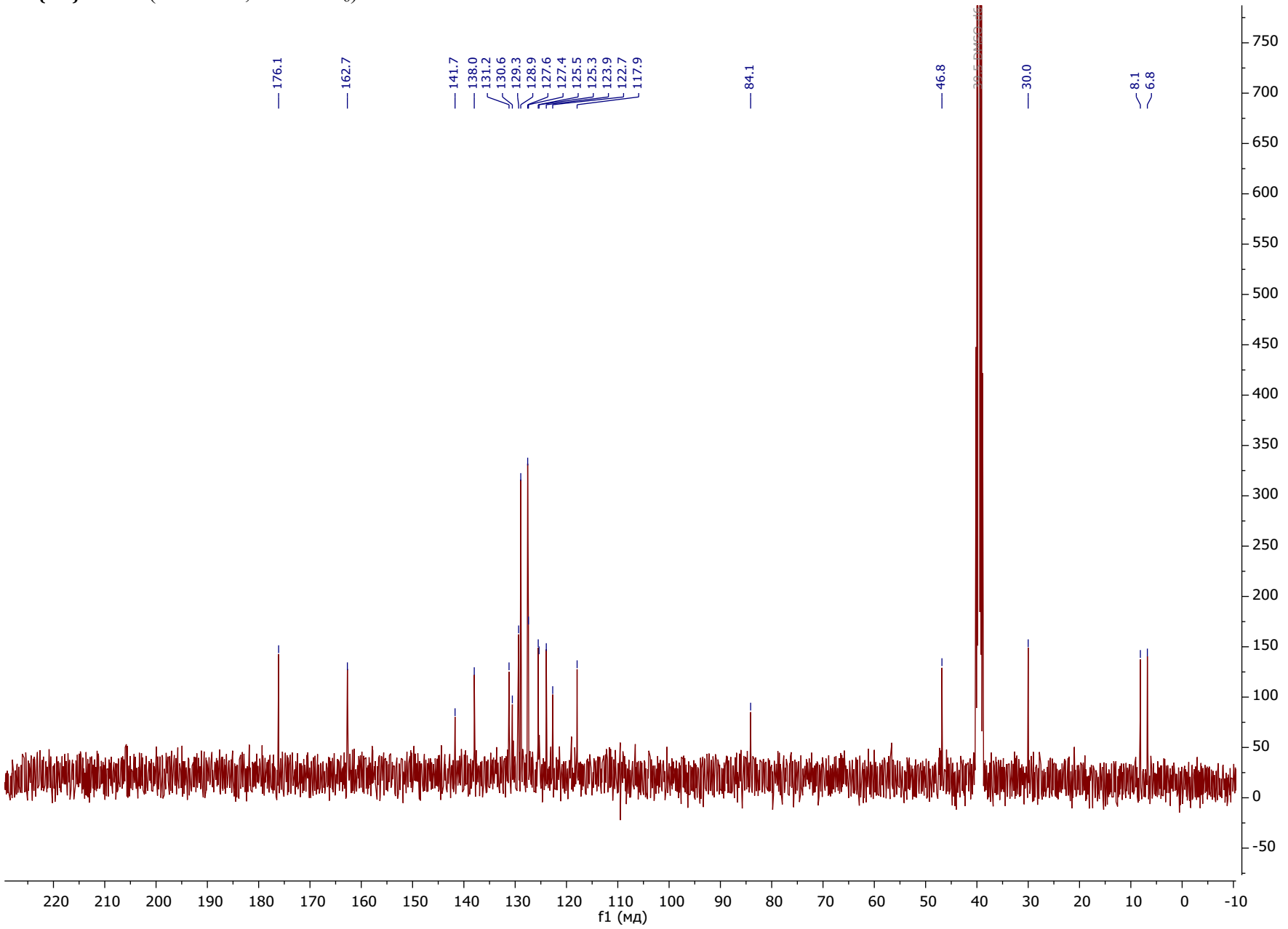
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3n**



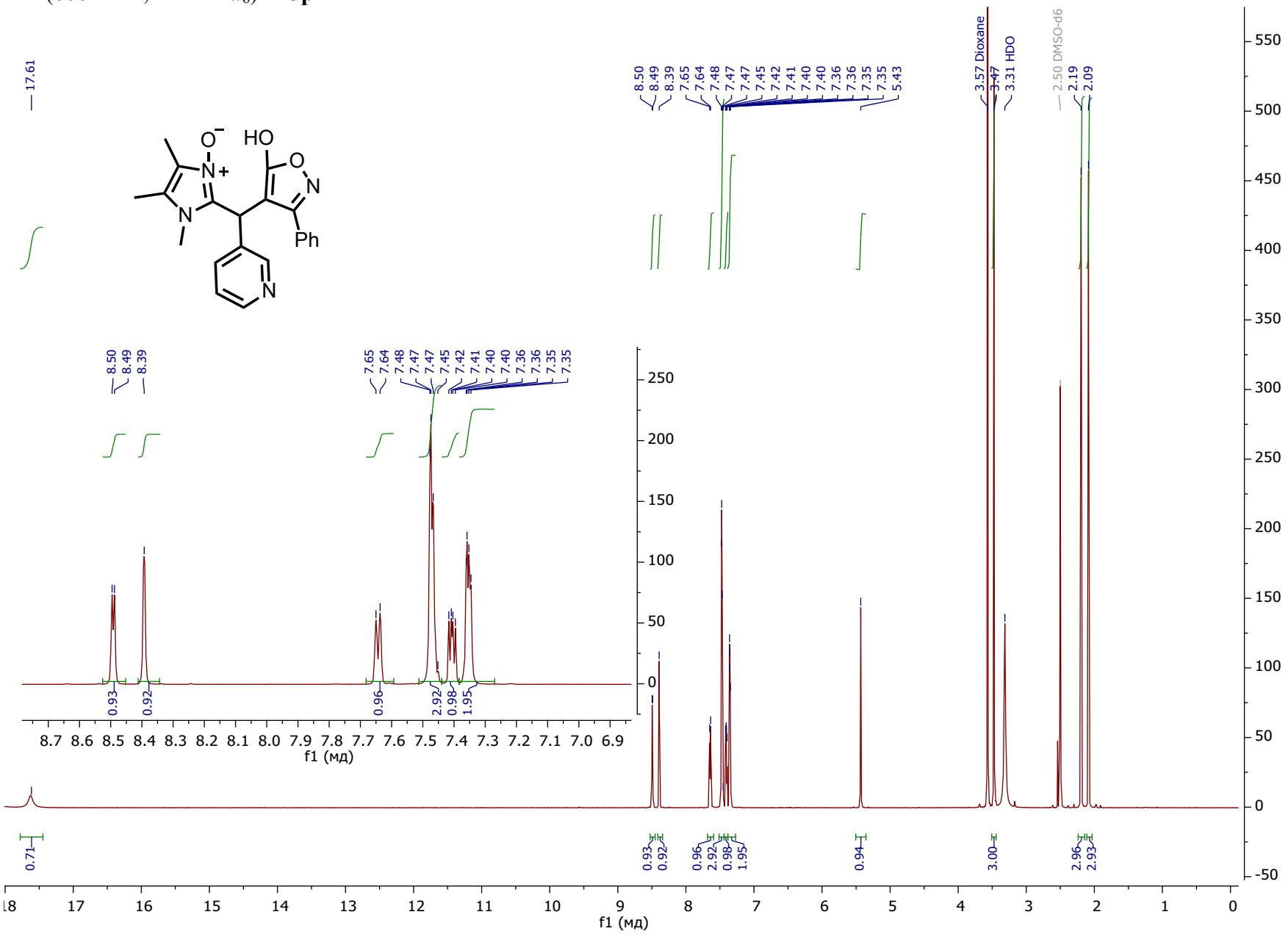
^{1}H NMR (400 MHz, DMSO-d_6) of **3o**



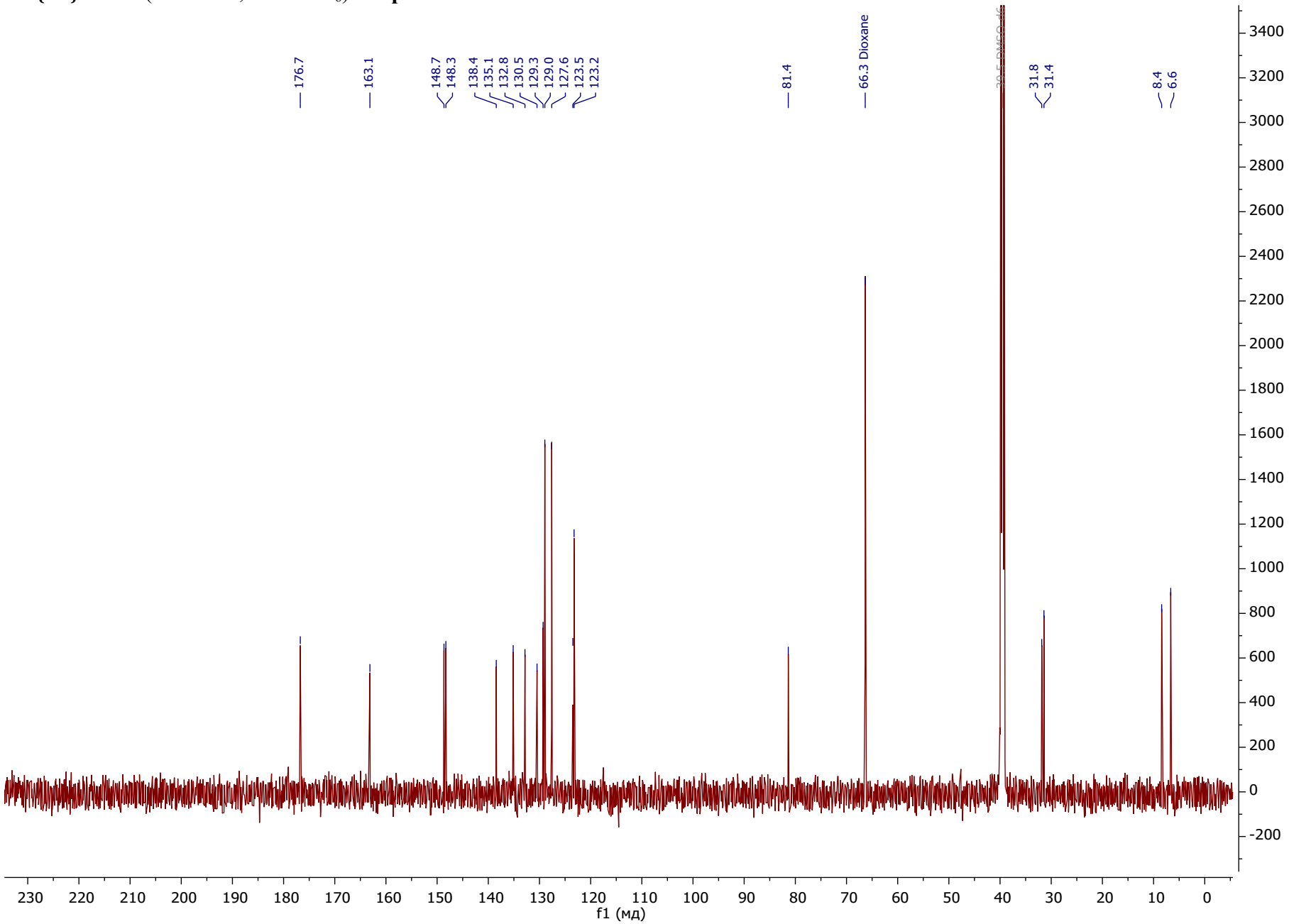
$^{13}C\{^{1}H\}$ NMR (101 MHz, DMSO-d_6) of **3o**



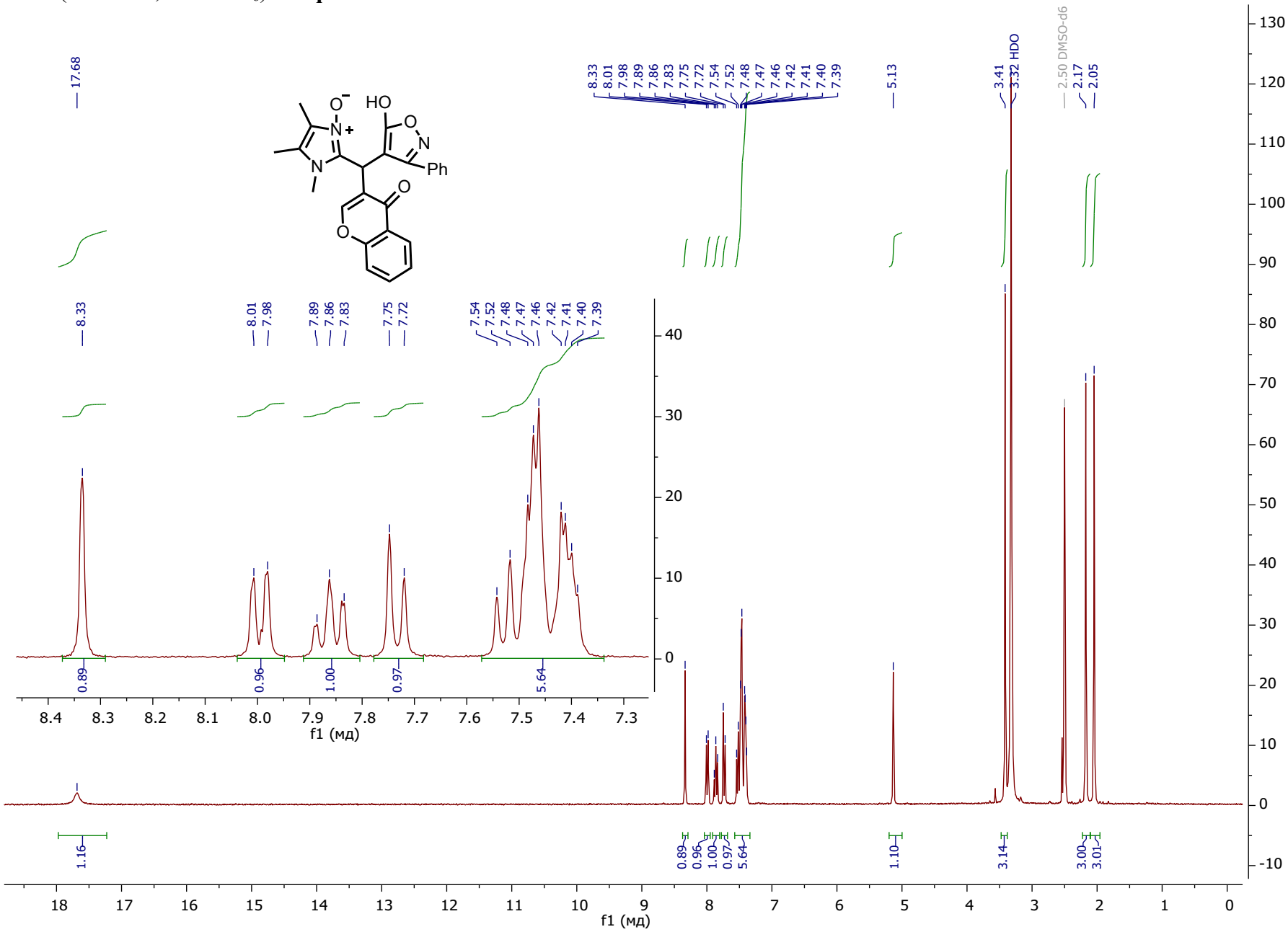
^{1}H NMR (600 MHz, DMSO-d_6) of **3p**



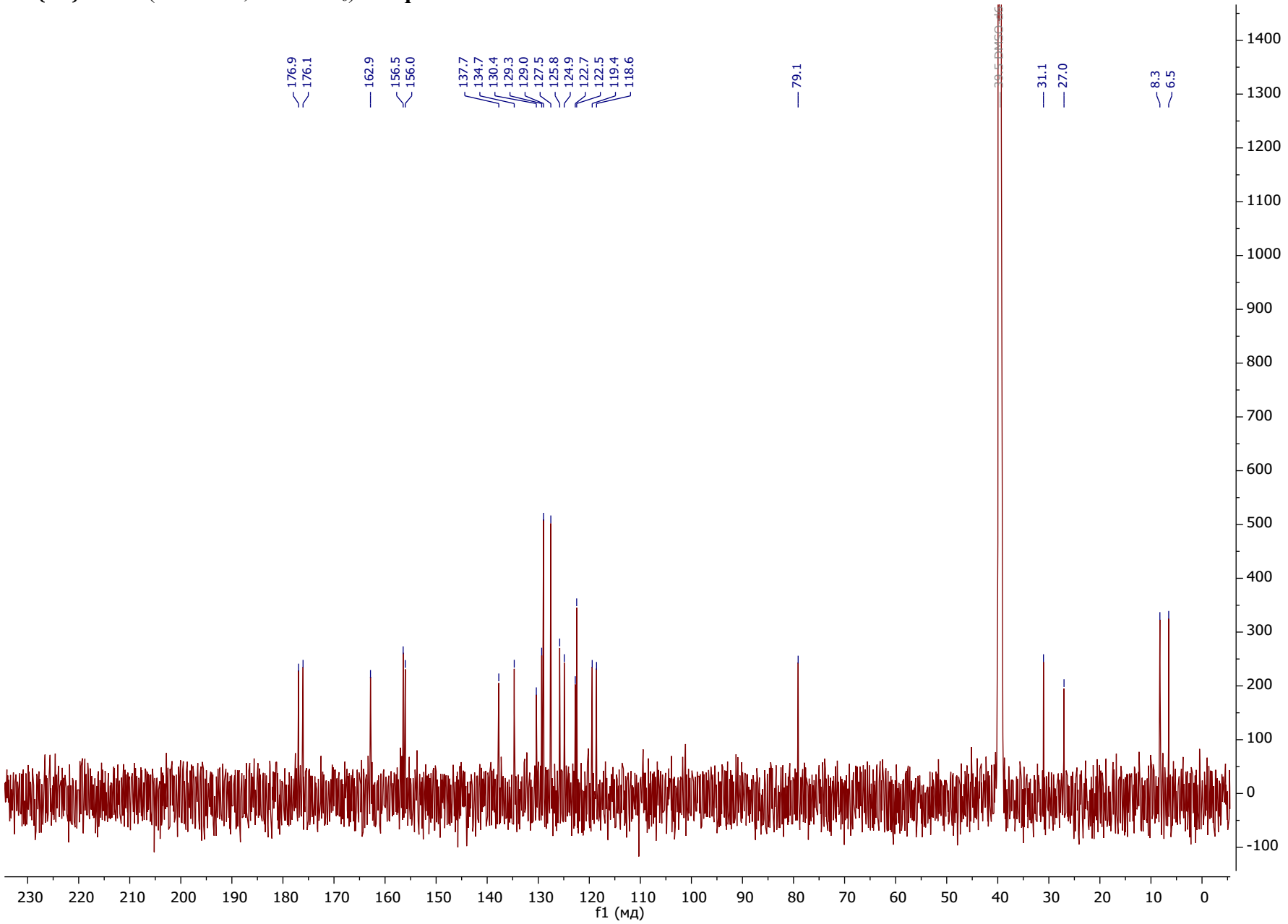
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3p**



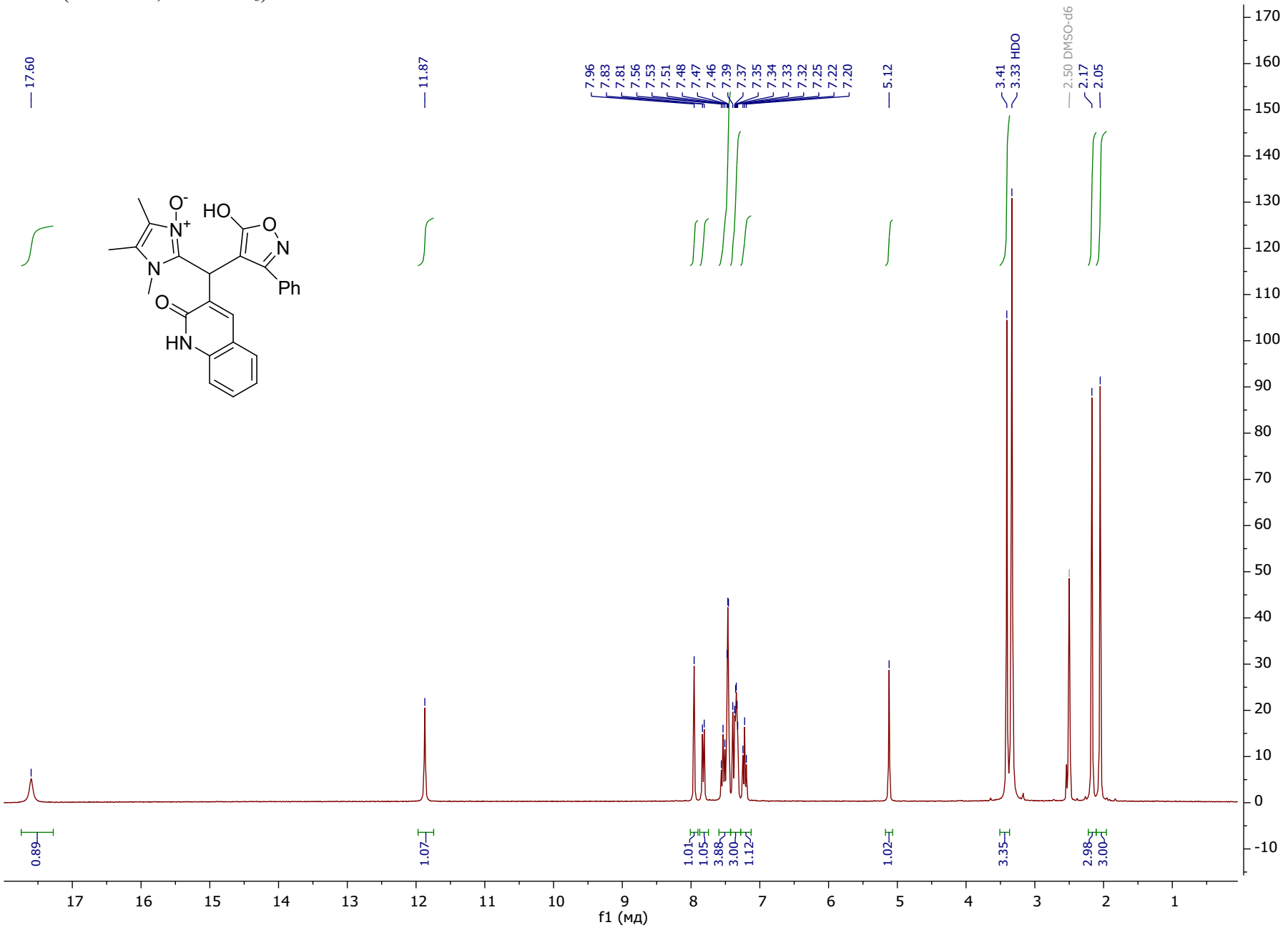
^{1}H NMR (300 MHz, DMSO-d_6) of **3q**



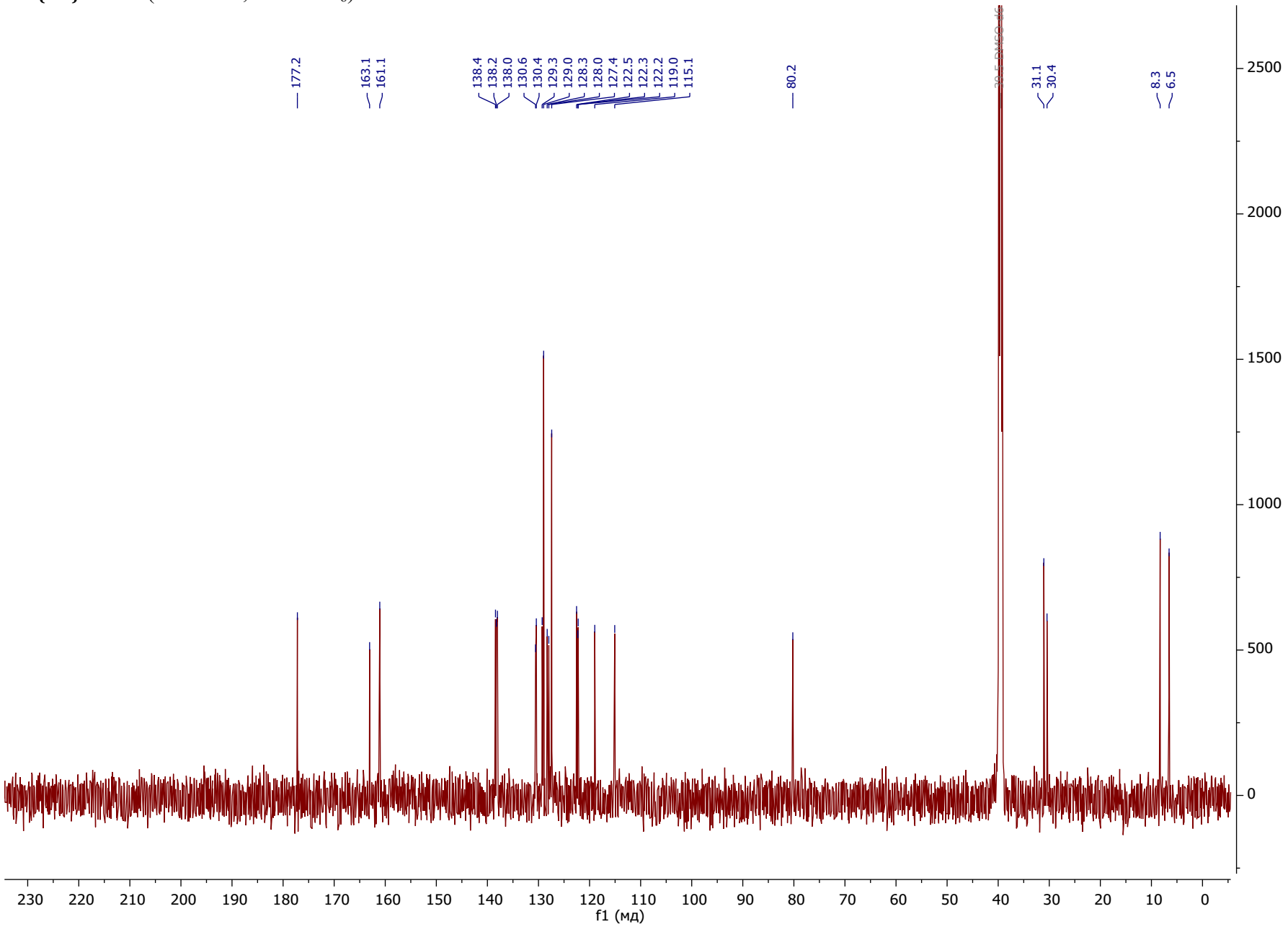
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3q**



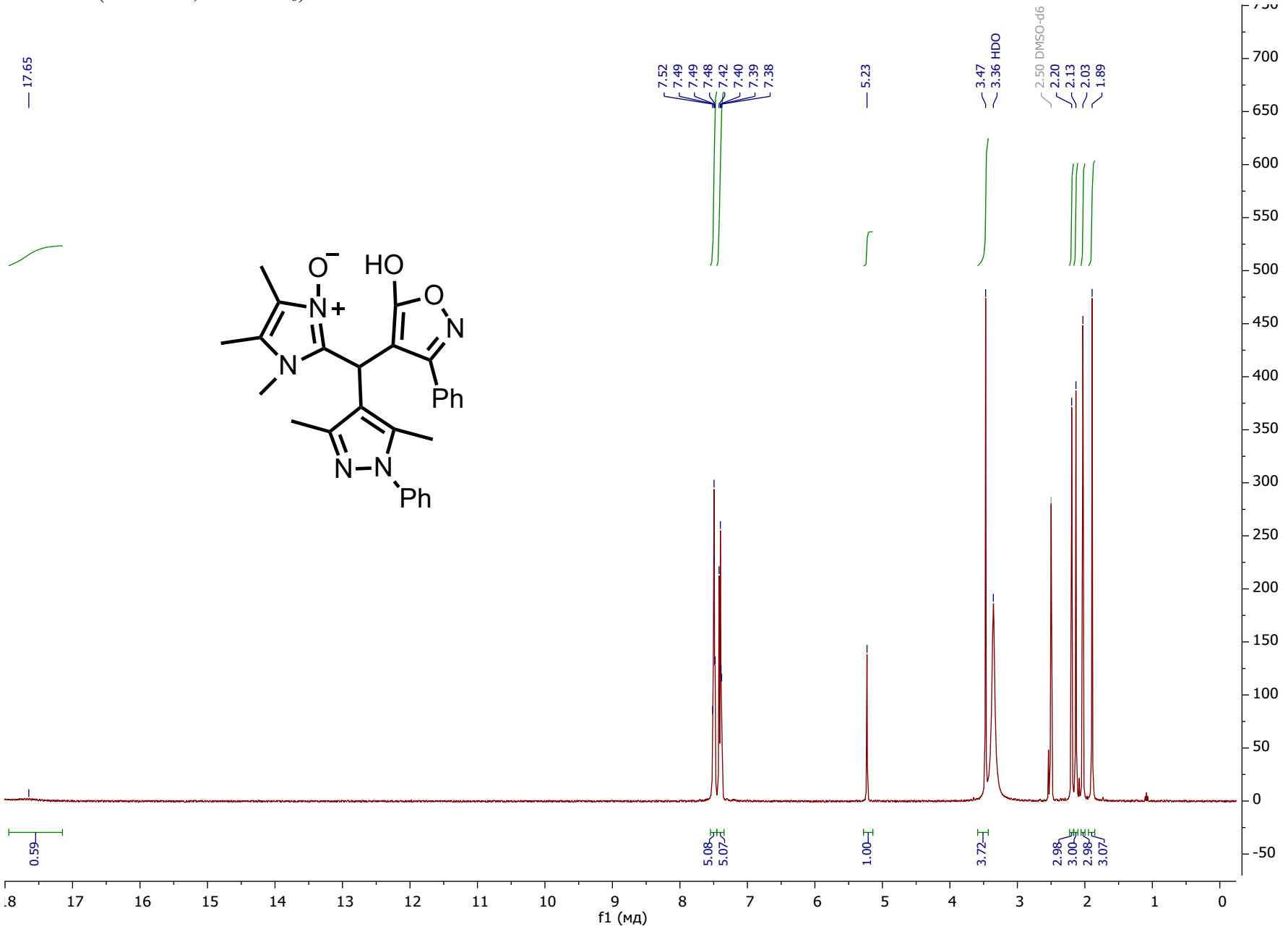
^{1}H NMR (300 MHz, DMSO-d_6) of **3r**



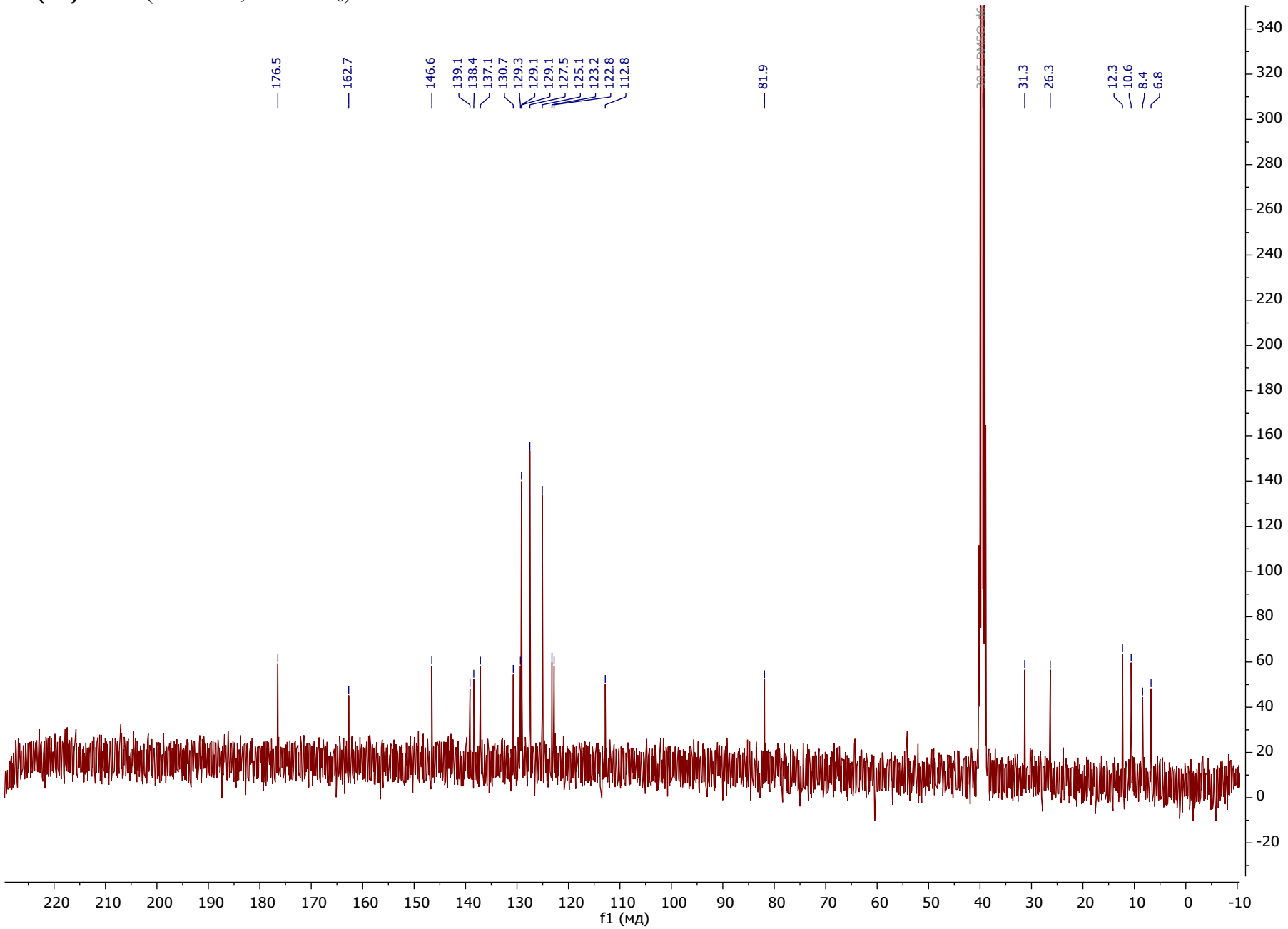
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3r**



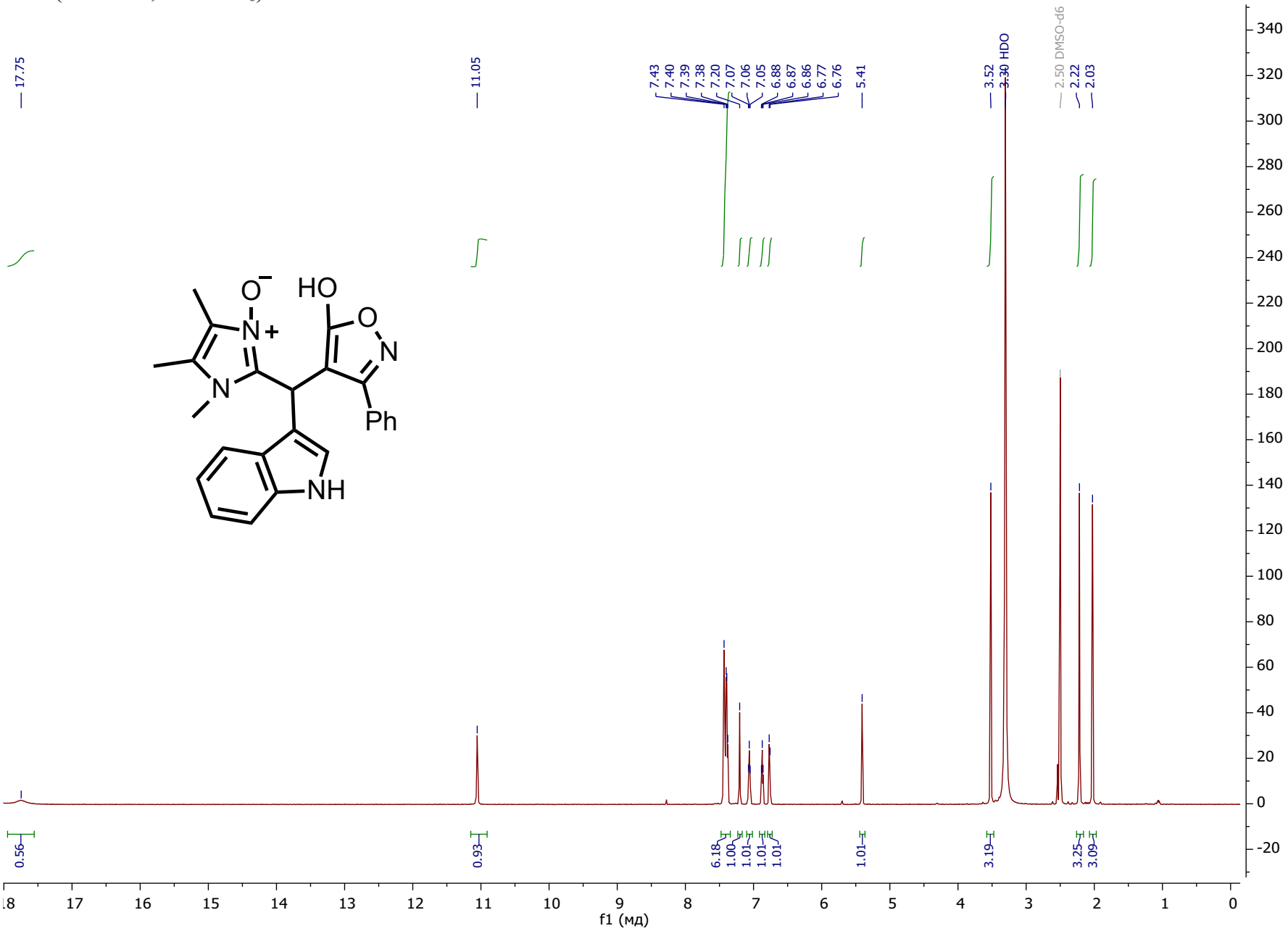
[1]H NMR (400 MHz, DMSO-d_6) of **3s**



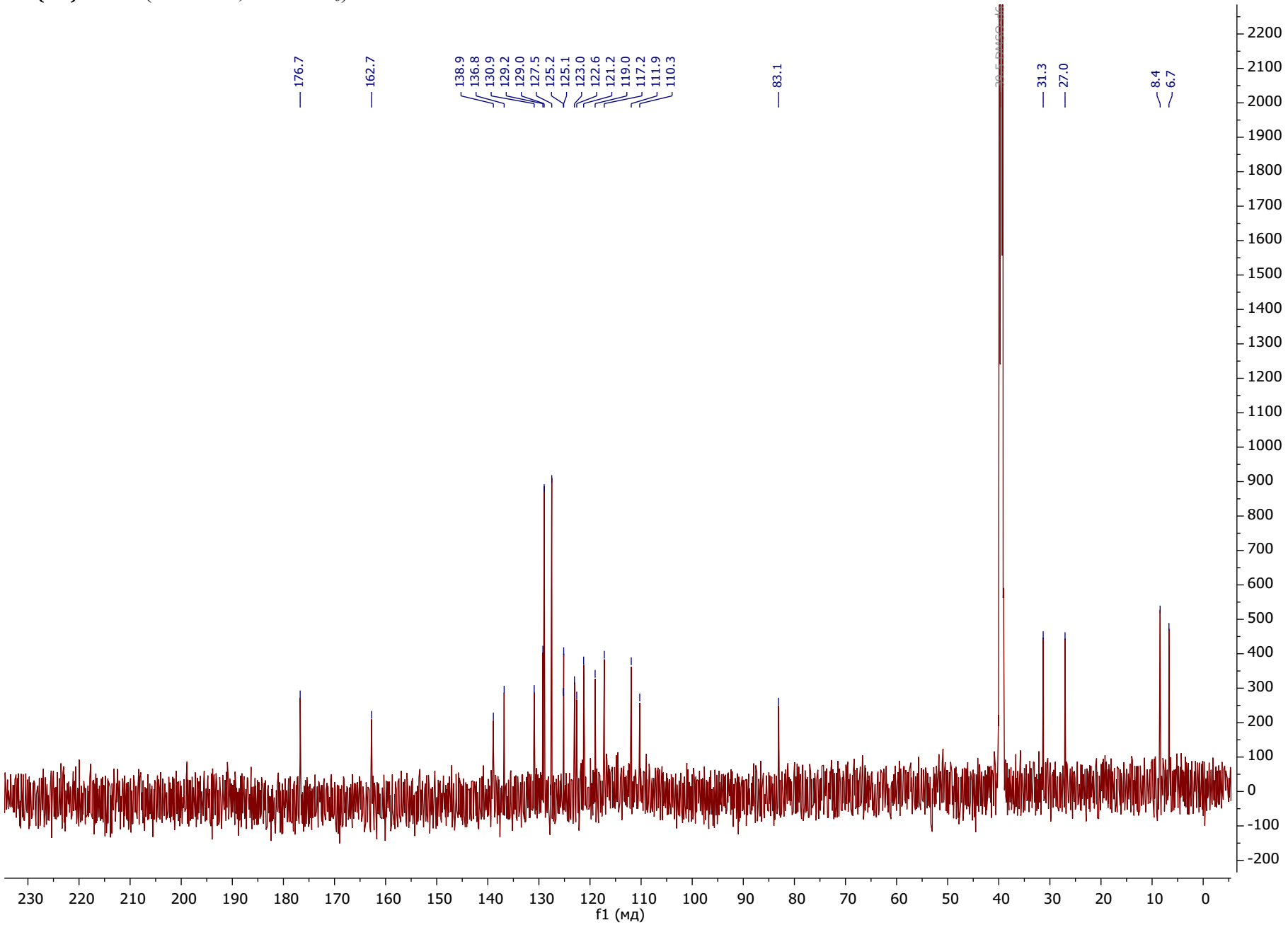
$^{13}C\{^{1}H\}$ NMR (101 MHz, DMSO-d_6) of **3s**



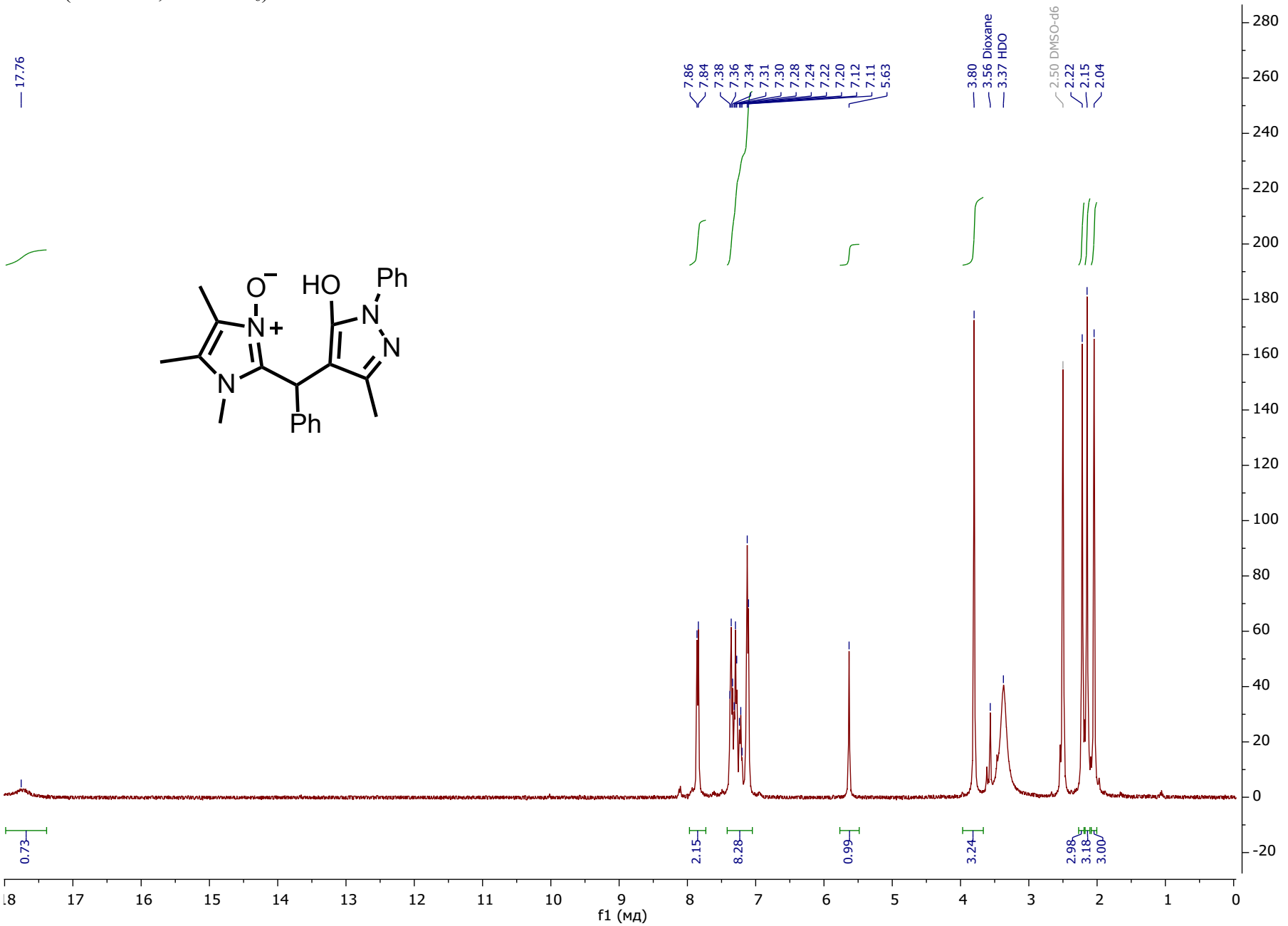
^{1}H NMR (600 MHz, DMSO-d_6) of **3t**



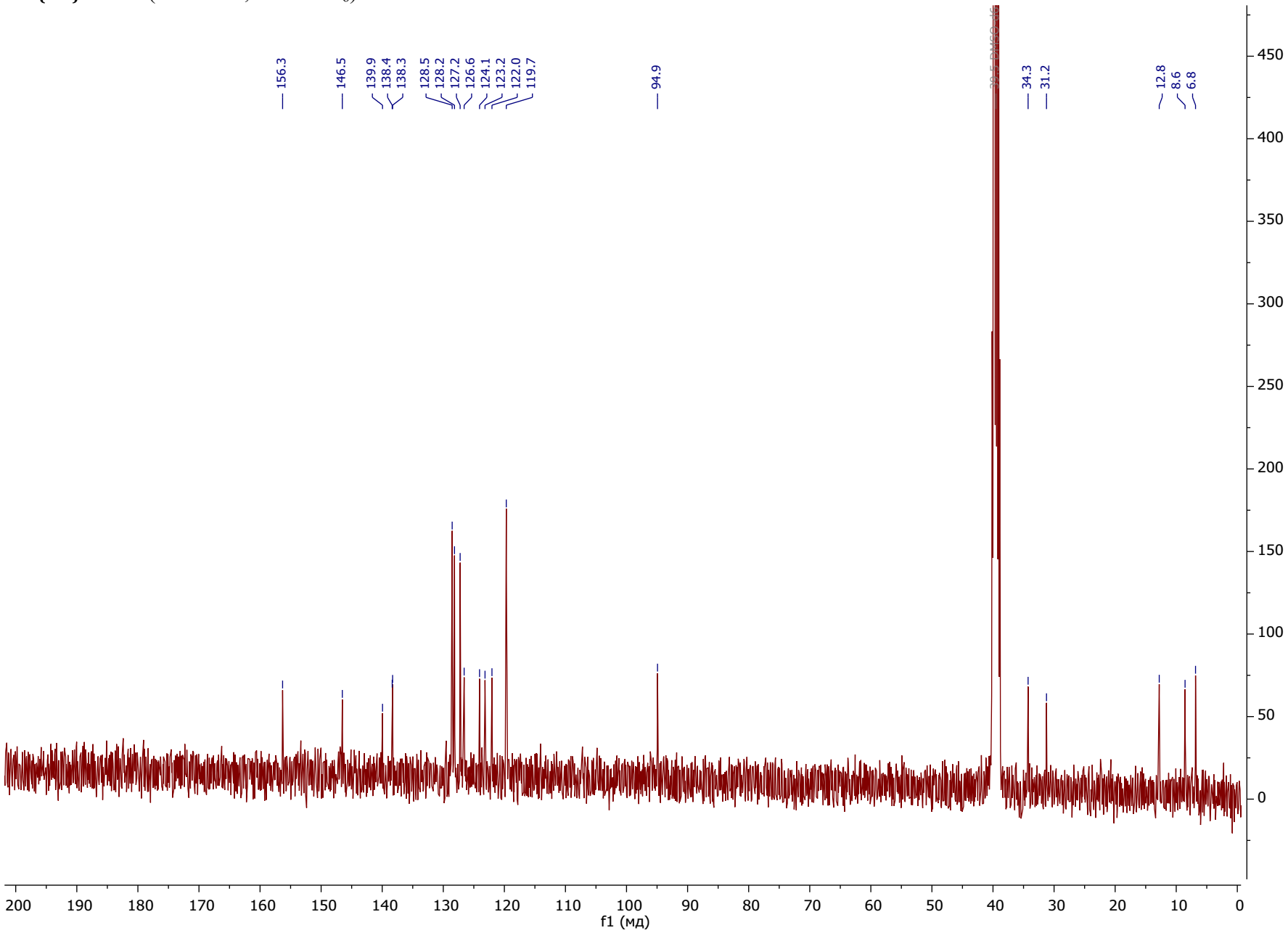
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **3t**



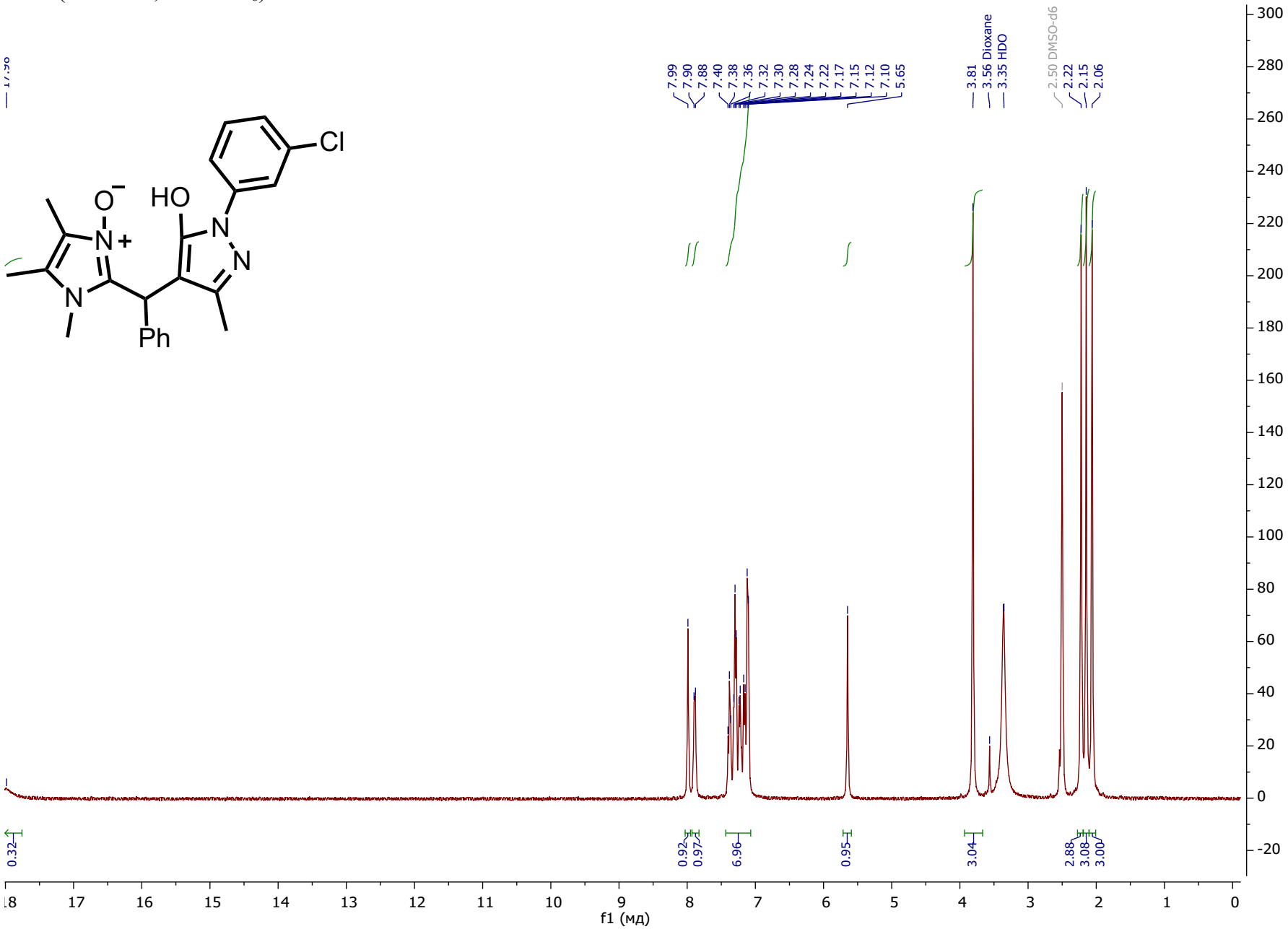
^{1}H NMR (400 MHz, DMSO-d_6) of **4a**



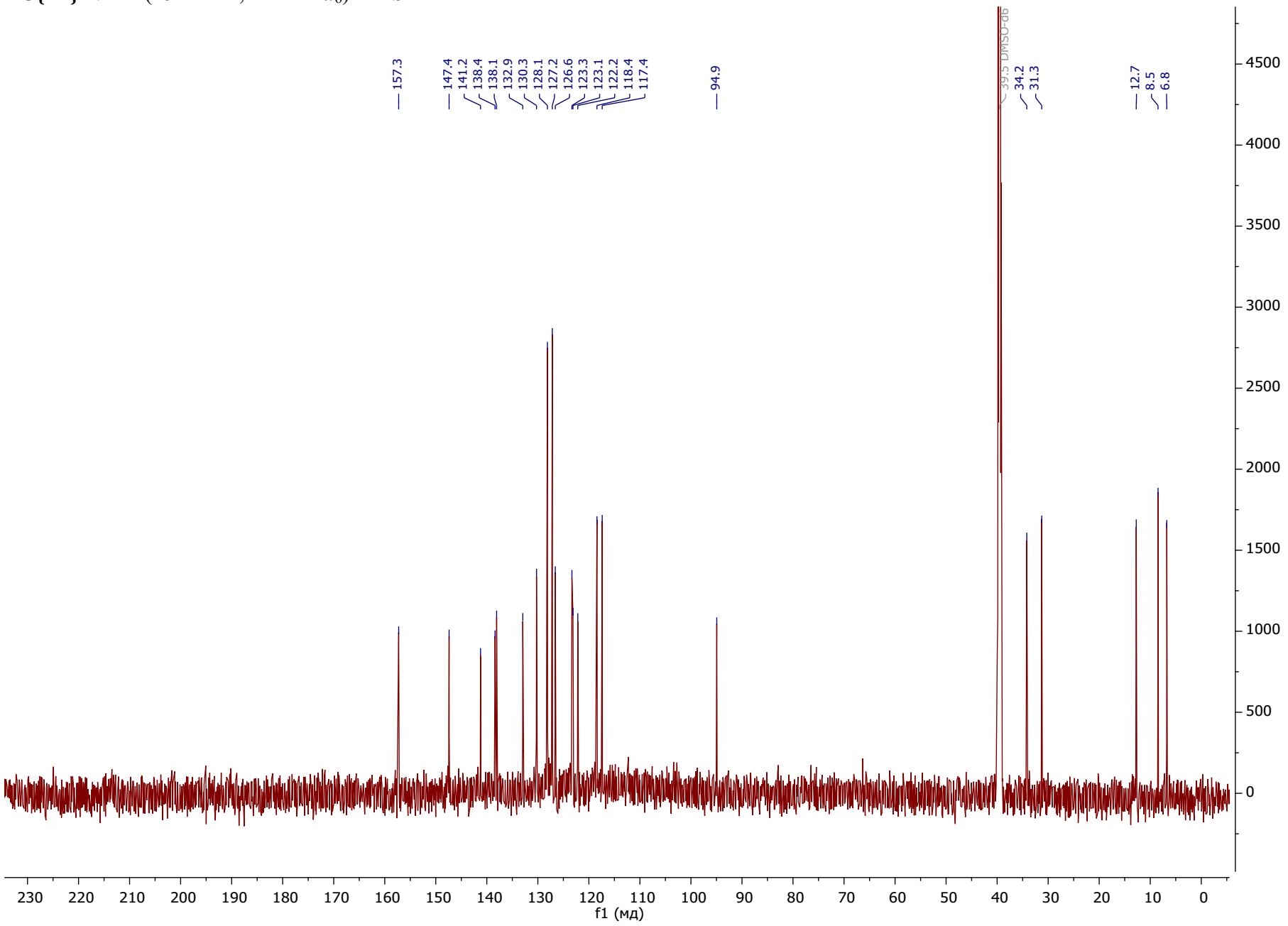
$^{13}C\{^{1}H\}$ NMR (101 MHz, DMSO-d_6) of **4a**



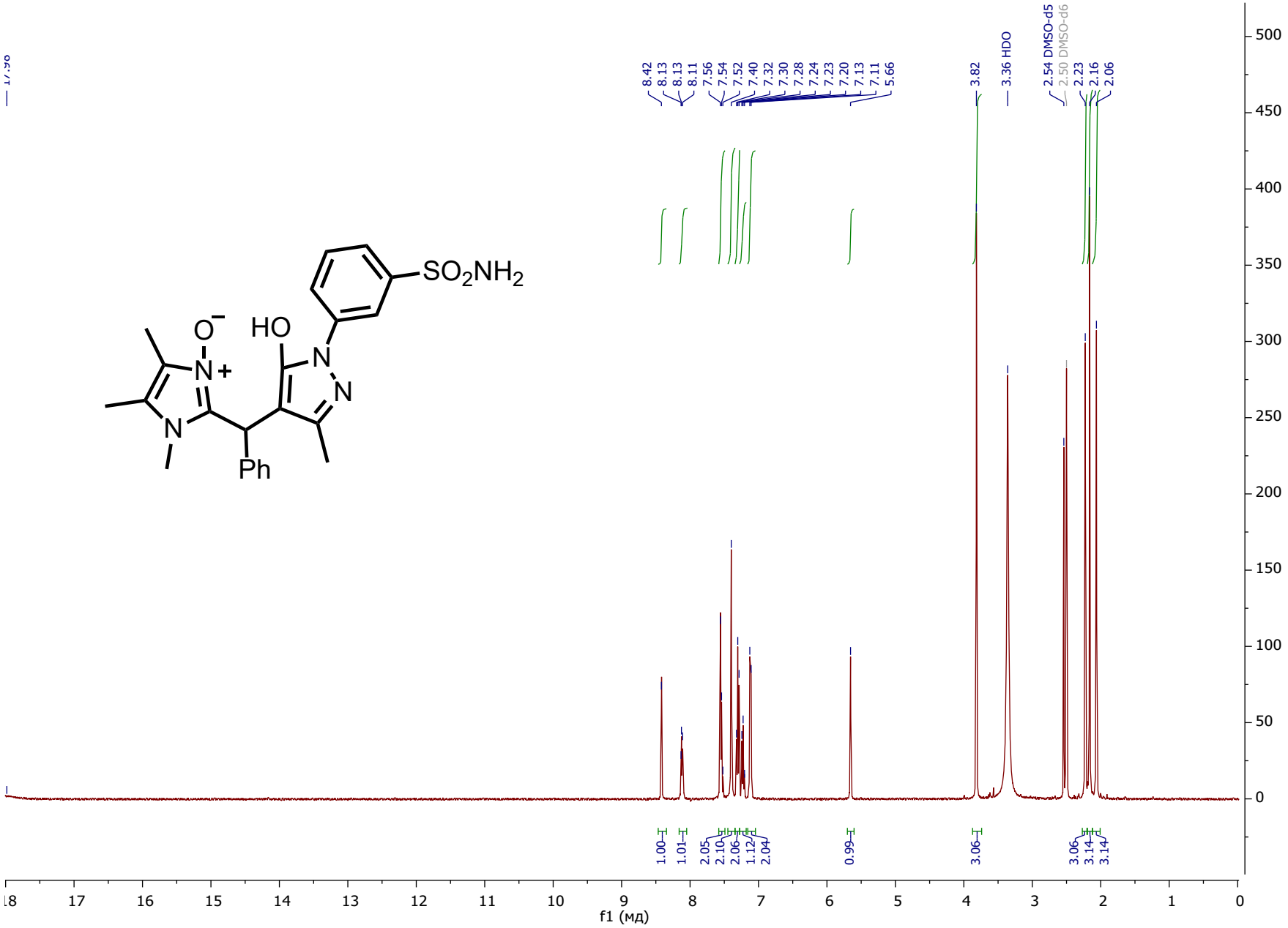
^{1}H NMR (400 MHz, DMSO-d_6) of **4b**



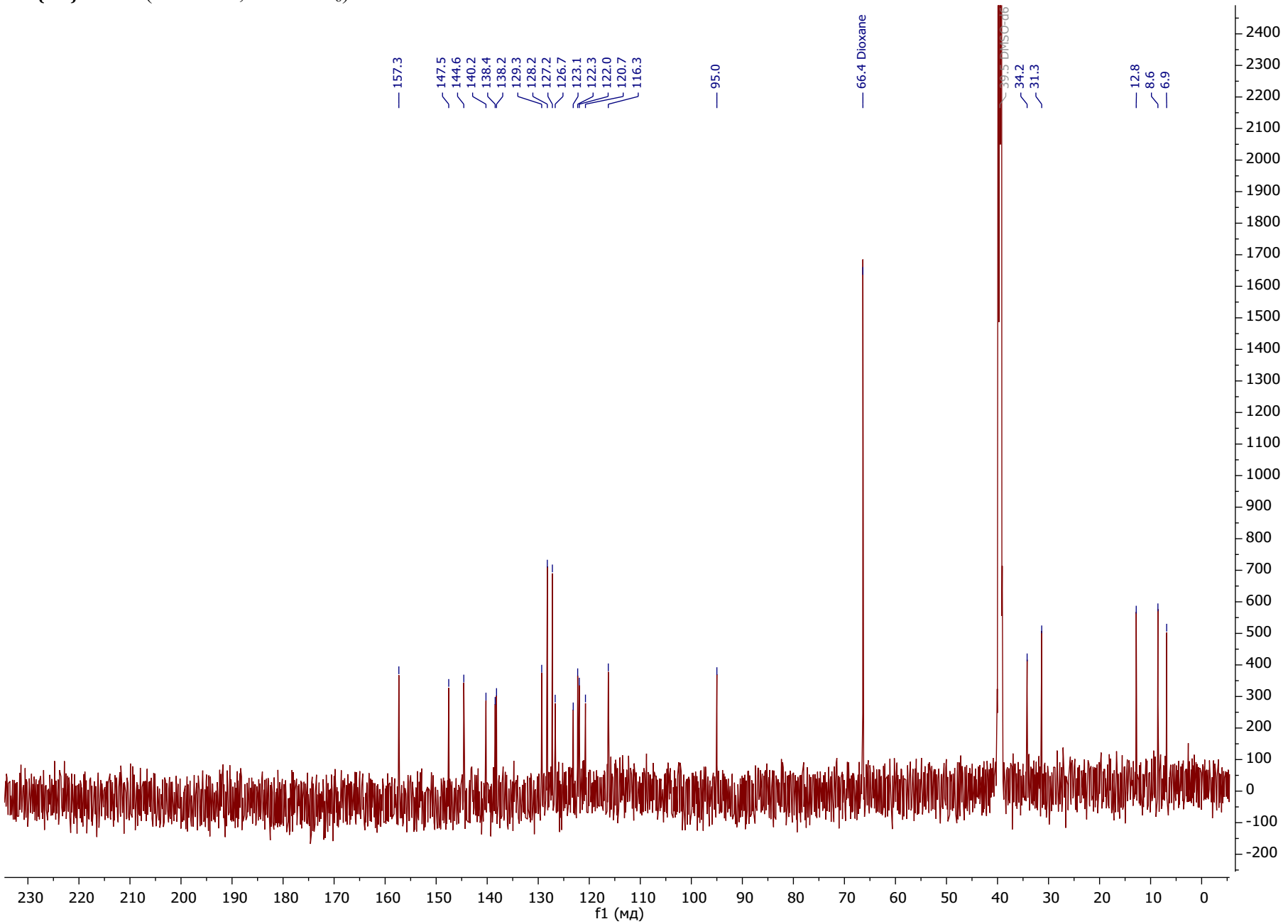
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **4b**



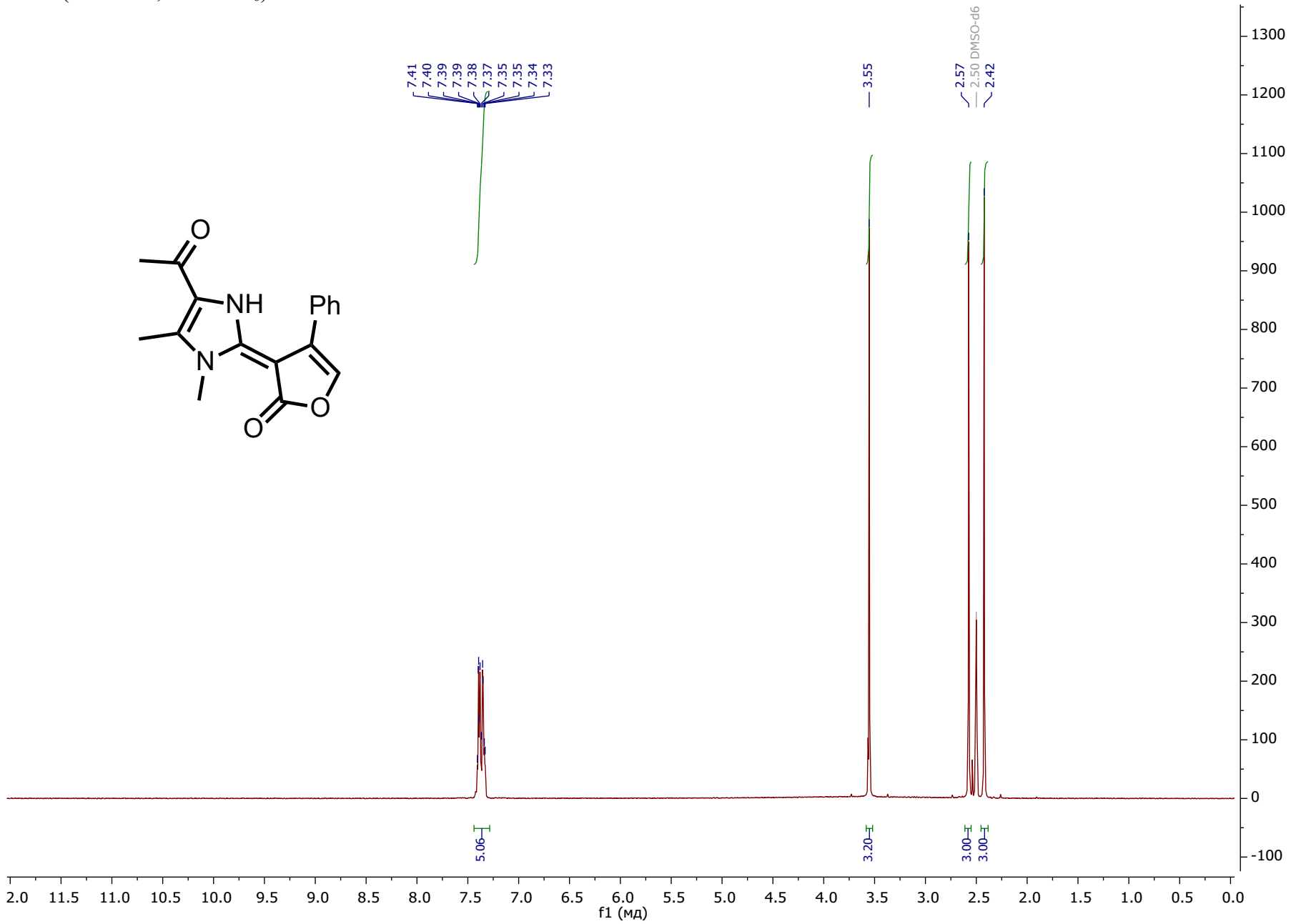
^{1}H NMR (400 MHz, DMSO-d_6) of **4c**



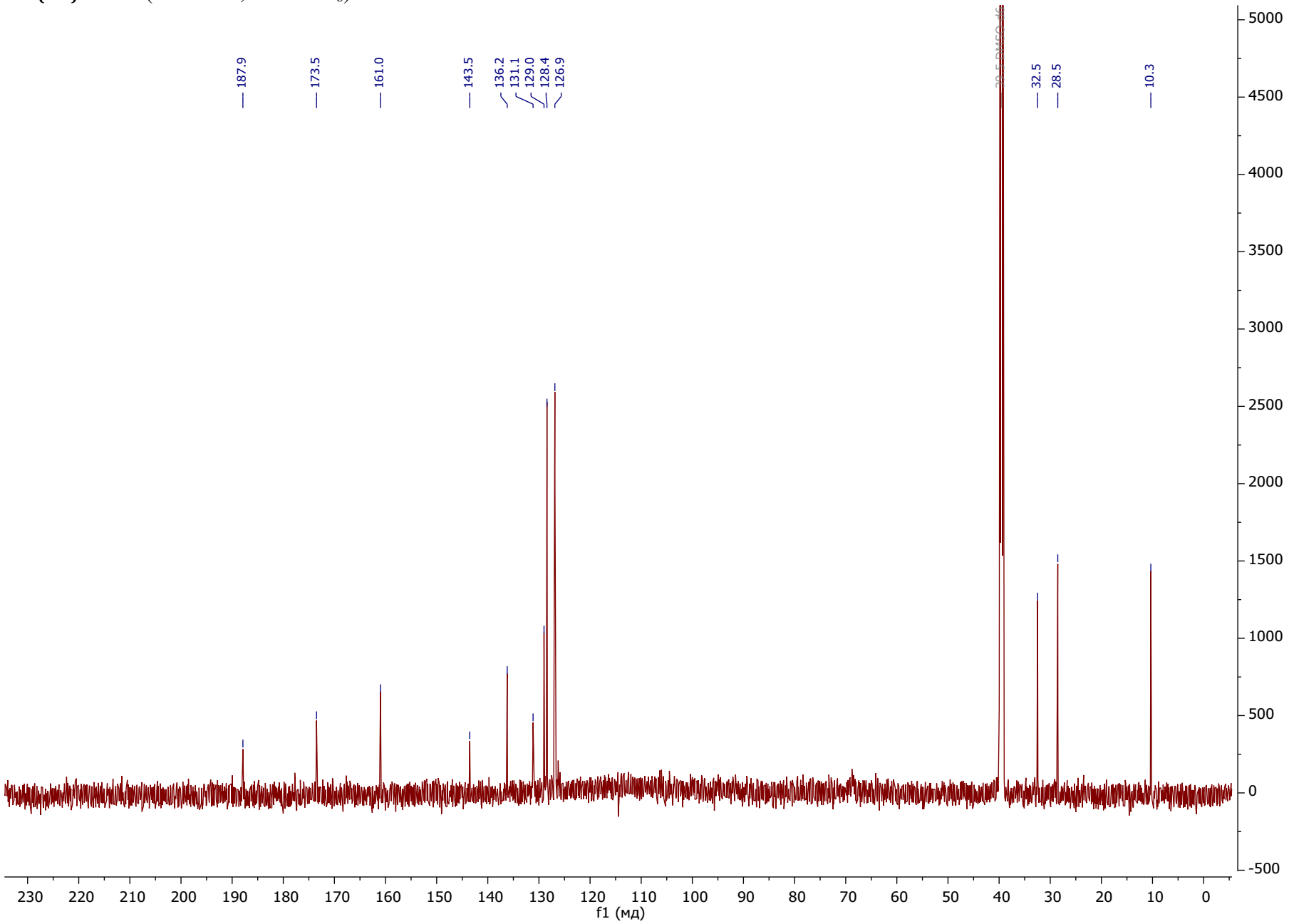
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **4c**



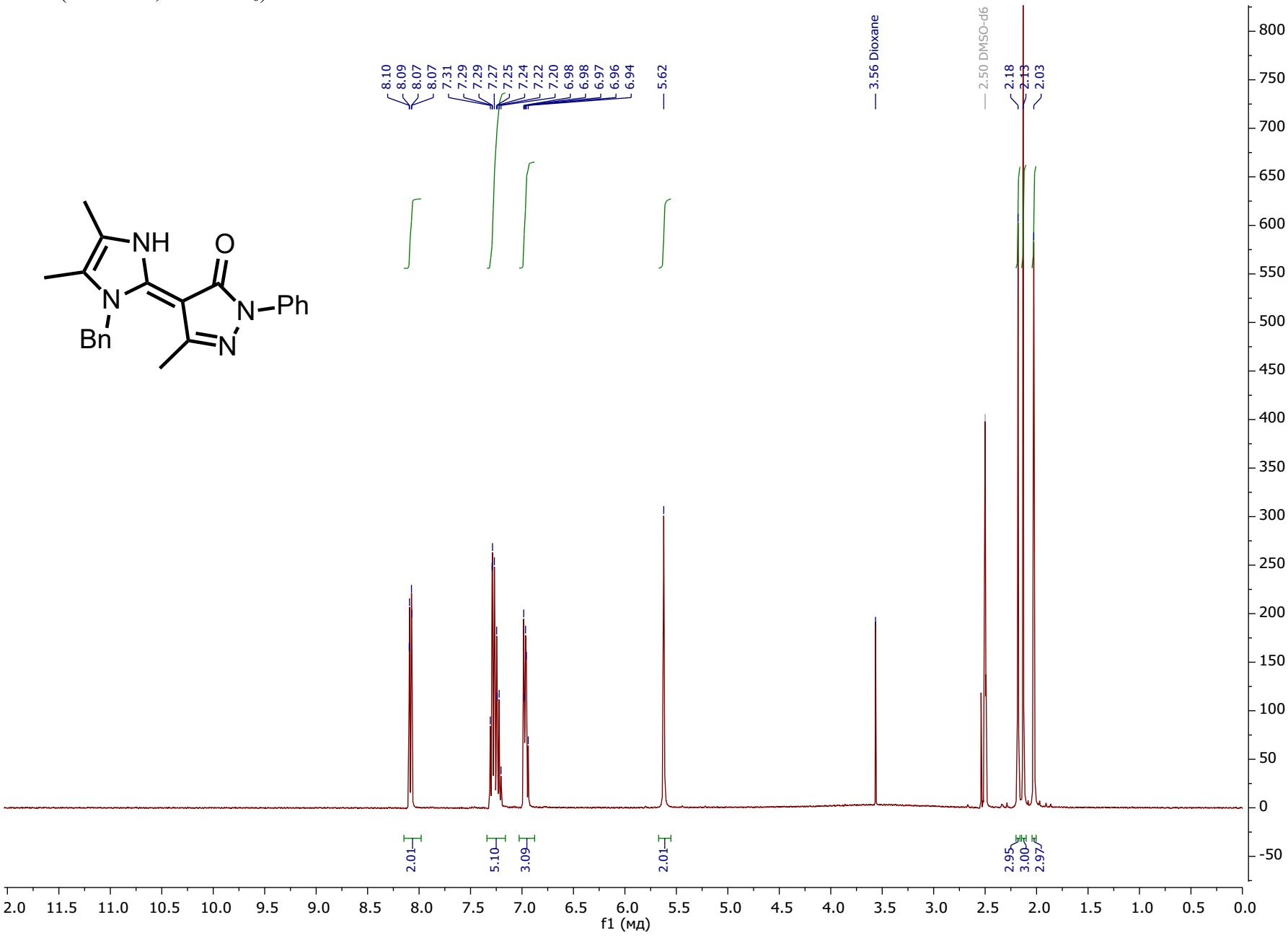
^{1}H NMR (400 MHz, DMSO-d_6) of **5**



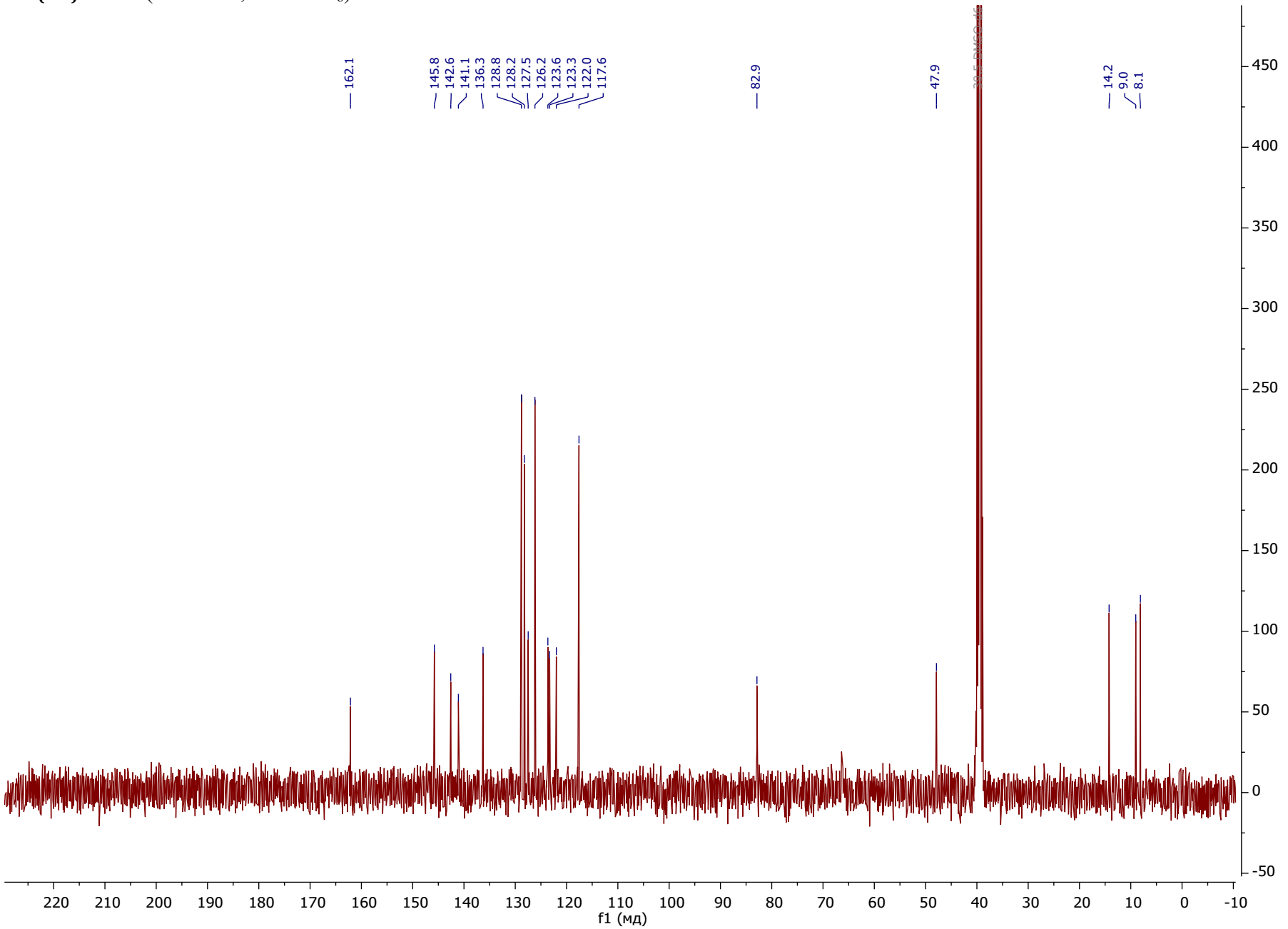
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **5**



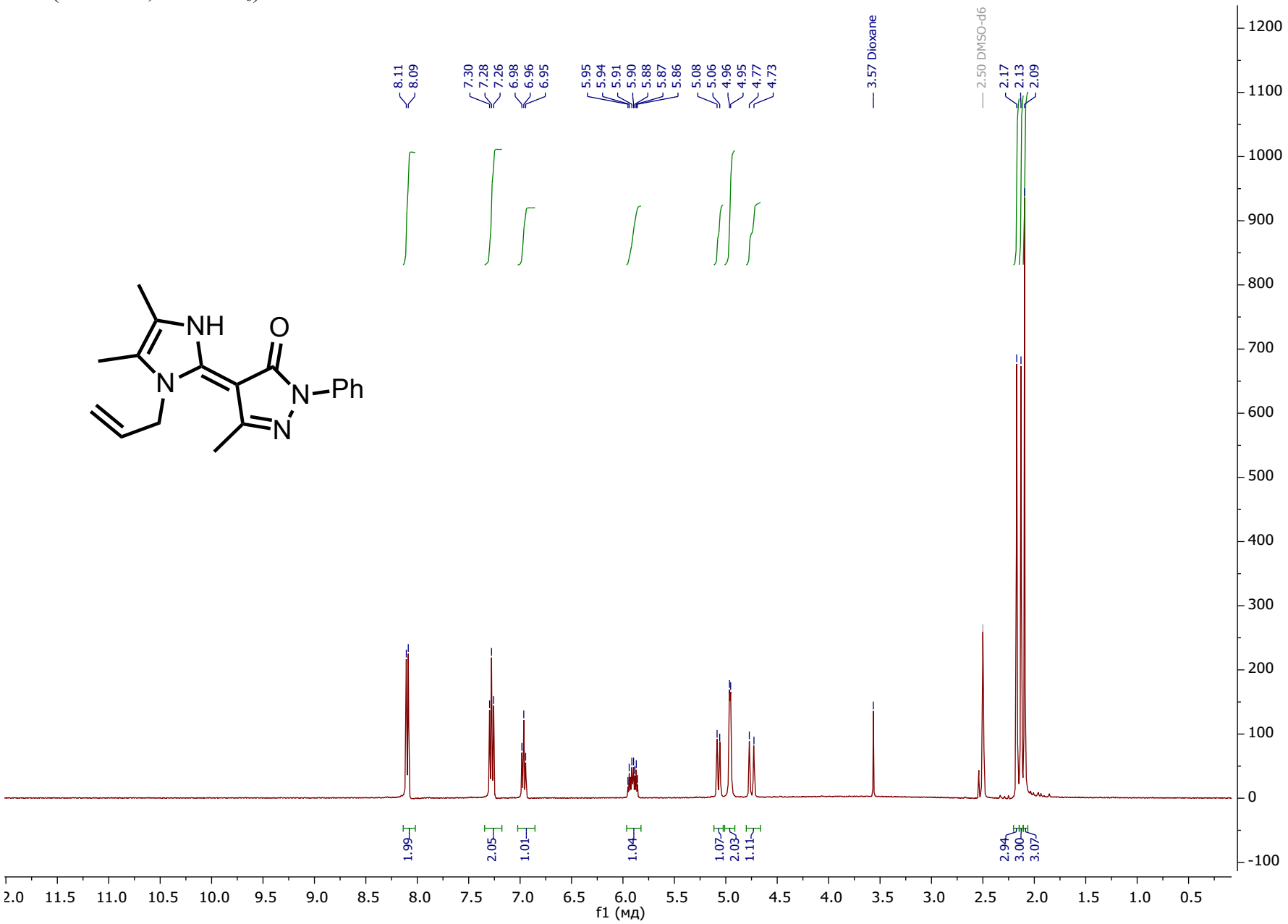
^{1}H NMR (400 MHz, DMSO-d_6) of **6a**



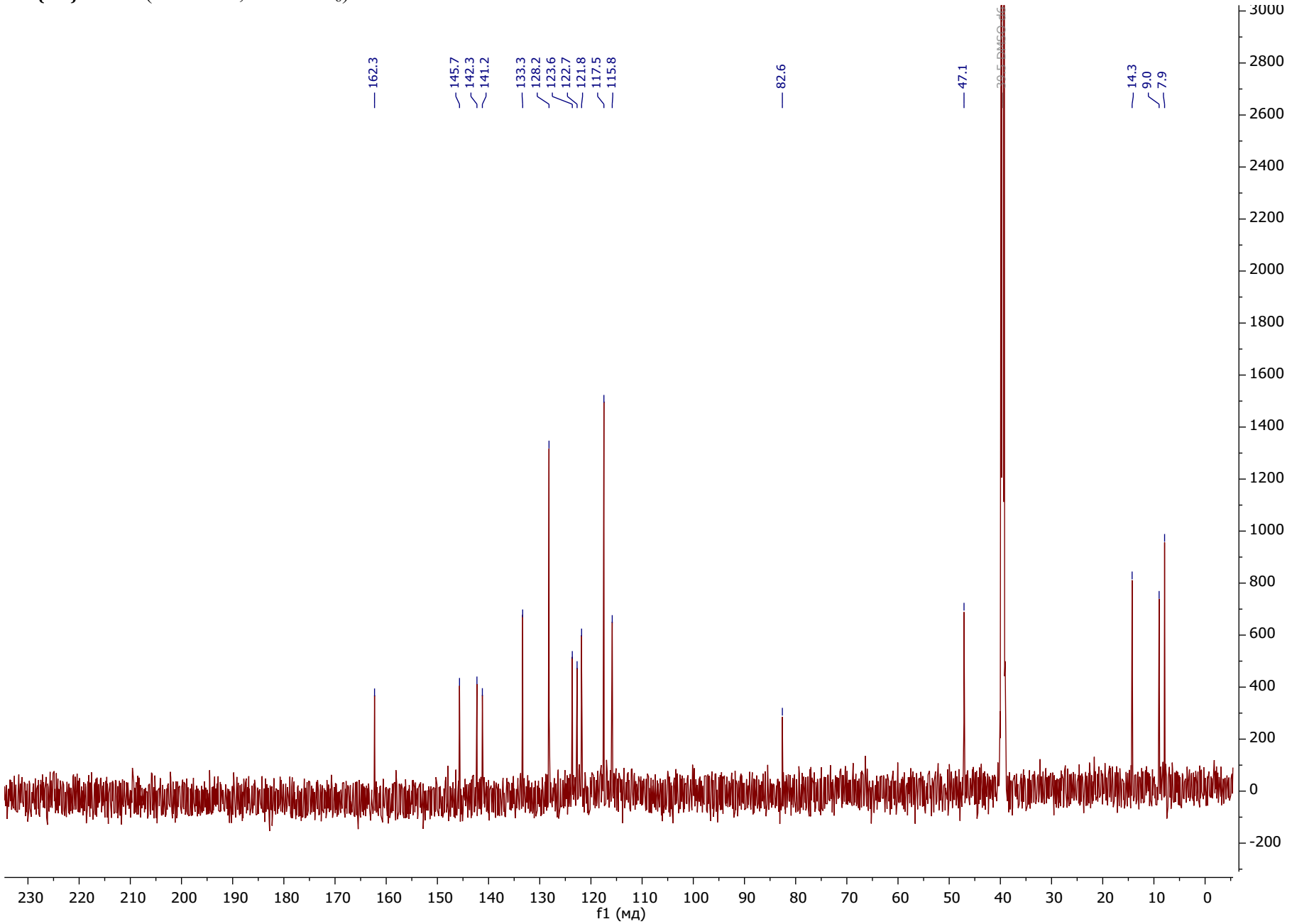
$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **6a**



^{1}H NMR (400 MHz, DMSO-d_6) of **6b**



$^{13}C\{^{1}H\}$ NMR (151 MHz, DMSO-d_6) of **6b**



X-ray crystallography. X-ray diffraction data for **5**, 3**c**, **3h** and **3j** were collected at 100 K with a Bruker Quest D8 CMOS diffractometer, using graphite monochromated Mo-Kα radiation (lλ = 0.71073 Å). Using Olex2,[1] the structures were solved with the ShelXT[2] structure solution program using Intrinsic Phasing and refined with the XL[3] refinement package using Least-Squares minimization against F^2 in anisotropic approximation for non-hydrogen atoms. Hydrogen atoms of NH group in **5** and those of OH groups in **3c**, **3h** and **3j** were located from difference Fourier synthesis while the positions of other hydrogen atoms were calculated, and they all were refined in isotropic approximation within the riding model. Crystal data and structure refinement parameters are given in Table 1. CCDC 2210062, 2210061, 2210576 and 2210575 contain the supplementary crystallographic data for **5**, **3c**, **3h** and **3j**, respectively.

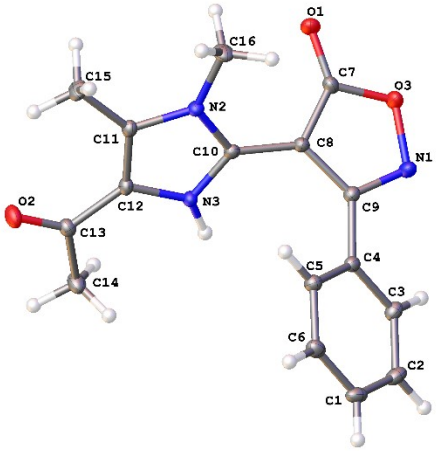


Fig. 1. General view of **5** in representation of atoms *via* thermal ellipsoids at 50% probability level.

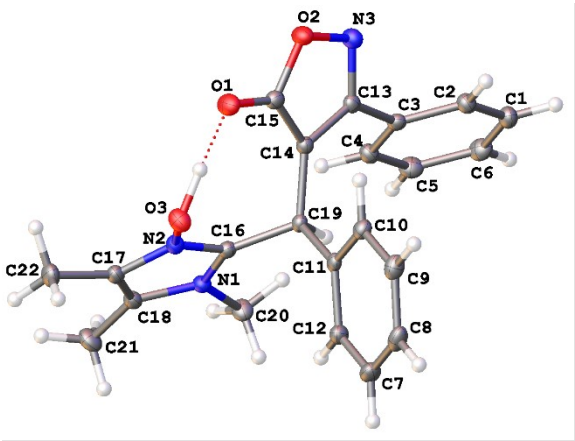


Fig. 2. General view of **3c** in representation of atoms *via* thermal ellipsoids at 50% probability level.

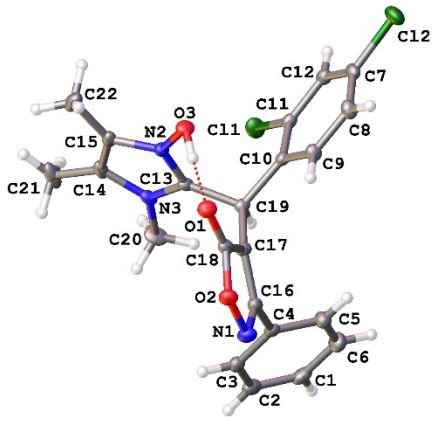


Fig. 3. General view of **3h** in representation of atoms *via* thermal ellipsoids at 50% probability level.

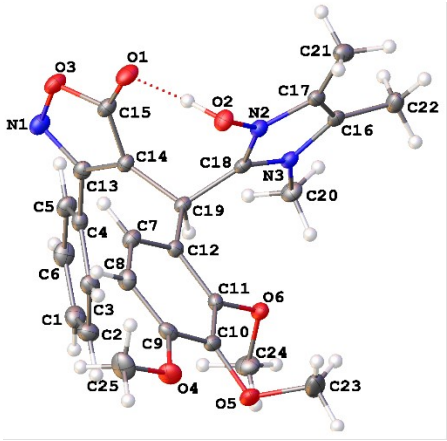


Fig. 4. General view of **3j** in representation of atoms *via* thermal ellipsoids at 50% probability level.

Table 1. Crystal data and structure refinement parameters for **5**, **3c**, **3h** and **3j**.

	5	**3c**	**3h**	**3j**
Empirical formula	$C_{16}H_{15}N_3O_3$	$C_{22}H_{21}N_3O_3$	$C_{22}H_{19}Cl_2N_3O_3$	$C_{25}H_{27}N_3O_6$
Formula weight	297.316	375.430	444.320	465.510
T, K	100	100	100	100
Crystal system	Orthorhombic	Monoclinic	Monoclinic	Triclinic
Space group	$Pna2_1$	$P2_1/n$	$P2_1/c$	P-1
Z	4	4	4	2
a, Å	8.0260(2)	8.1378(1)	7.7044(1)	8.3902(2)
b, Å	18.0409(4)	24.1686(4)	12.9589(2)	11.0278(2)
c, Å	10.1247(2)	10.0882(2)	20.3448(3)	14.1048(3)
□, °	90	90	90	70.487(1)
□, °	90	111.523(1)	92.514(1)	84.727(1)

□, °	90	90	90	72.340(1)
V, Å^3	1466.02(6)	1845.79(5)	2029.28(5)	1172.06(4)
D_{calc} (g cm^{-1})	1.347	1.351	1.454	1.319
□, см$^{-1}$	0.95	0.92	3.5	0.95
F(000)	624	792	920	492
2□$_{max}$, °	58	58	58	58
Reflections measured	27619	24713	26626	15851
Independent reflections	3887	4899	5389	6209
Observed reflections [I > 2σ(I)]	3337	4028	4673	4844
Parameters	203	256	274	313
R1	0.0304	0.0421	0.0335	0.0450
wR2	0.0729	0.1092	0.0865	0.1111
GOF	1.022	1.024	1.027	1.019
$\Delta\rho_{max}$/ $\Delta\rho_{min}$ (e Å^{-3})	0.225/-0.230	0.396/-0.357	0.370/-0.302	0.302/-0.291

References

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2 G. M. Sheldrick, *Acta Crystallogr. Sect. A Found. Adv.*, 2015, **71**, 3–8.

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